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with the author's regard.*

ON

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## DEFECTS IN THE APPARATUS

GENERALLY USED FOR THE DETERMINATION OF

# BISULPHIDE OF CARBON

IN

## COAL GAS,

AND DESCRIPTION OF A

## NEW APPARATUS FOR ITS ESTIMATION.

BY

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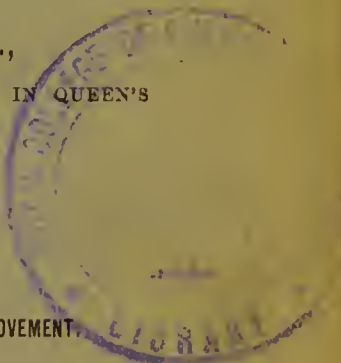
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ON  
DEFECTS IN THE APPARATUS,

&c. &c.

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It is, I think, very generally admitted that the question of the determination of the exact proportion of sulphur in the bisulphide of carbon form in coal gas assumes every day more importance. Increased observation which I have been afforded during the past few months has further contributed to prove to me the serious defects inseparable from the general arrangement of apparatus, and the usual procedure in manipulation that are at present in use for that purpose. While our processes for the estimation of sulphur remain in this condition, of what reliable value are statements to the effect that, by the adoption of this or that process, nine-tenths of the bisulphide of carbon compounds have been absorbed from the gas subjected to its action, when the apparatus at our disposal, by which we analyze the gas, possesses defects so serious that, unless extraordinary precautions have been taken, sulphur to the extent of 20 to 30 per cent. may probably, and almost certainly, have escaped determination! I have made many attempts, in consequence of this state of our analytical methods, to improve, if not to make quite perfect, an apparatus, together with the subsequent chemical operations of analysis, which should still be convenient in application while effecting this end. The imperfections in the apparatus furnished by Dr. Letheby were alluded to by myself some months since (p. 657 of the JOURNAL OF GAS LIGHTING, for Sept. 5, 1865). In speaking, upon that occasion, of the apparatus devised and recommended by him for ascertaining the amount of sulphur existing in the gas as bisulphide of carbon and such analogous combinations of sulphur, I took occasion to remark—“It is very well known that the apparatus described by Dr. Letheby is not without certain defects when brought into requisition for this purpose.” In this paper I have to bring before the notice of those interested in the chemistry of coal gas certain points of great importance, which are entirely associated with this question of sulphur determination. One is in regard to a new and very easily adapted modified arrangement of the apparatus I formerly used,\* by which I entirely prevent what has hitherto been considered and admitted by all who have studied the subject to be a

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\* See JOURNAL OF GAS LIGHTING, vol. xiv., p. 658.

source of error peculiar to it by no means insignificant. I here refer to the augmentation of the quantity of sulphur in the first place burned into sulphurous acid, and subsequently detained in combination with the ammoniacal condensed products in the receivers L M (see p. 658 of vol. xiv. of JOURNAL OF GAS LIGHTING); by the retention there likewise of the sulphurous portions always, especially in towns, forming a constituent in more or less proportion of atmospheric air. Now, in the absence of any data in respect to the quantity of these sulphur compounds in the air of various localities, or supposing that their proportions were known, the uncertainty and difficulty that must attend any attempt to ascertain the volume of air which, during the performance of an experiment, would necessarily pass up the trumpet-tube *o w*, and thence through the receivers M L, finally passing out at *y*, it is clear that, while the apparatus, with these defects inherent in its construction, whether the sulphite of ammonium at length obtained in the receivers does not contain a proportion of sulphur always in excess of that which actually existed in the tested volume of gas, must be, of course, a matter of great uncertainty. Barlow and Ellissen (JOURNAL OF GAS LIGHTING, vol. xiii., p. 713), in their report to the directors of the Chartered, City of London, and Great Central Consumers Companies, made in July, 1864, observe in reference to this subject:—"Nor are the processes at present employed for determining the quantity of sulphur so free from sources of error as to justify us in concluding that the whole (sulphur) is arrested by any of them. It is, however, certain that none of them will indicate the presence of a *greater* quantity of sulphur than really exists, if *proper precautions be taken to purify the air necessary for the combustion of the gas*, without which precaution the sulphur present in the air would tend to augment the quantity indicated as existing in gas. In Dr. Angus Smith's investigations on 'The Air of Towns,' the per centage of the volume of sulphurous acid present in the air of Manchester is stated to vary from 0.0011088 to 0.0000264, quantities which, though small, are quite sufficient to be appreciable in the products of the combustion of gas, and this per centage will be greater still in close rooms in which the gas is burnt." It must at the same time, however, be admitted as quite true, that in employing the apparatus defined by Dr. Letheby two sources of error, which have not hitherto been avoided, to some extent counteract each other in a manner tending to produce greater accuracy than would otherwise be attained. Thus it is always found by those who have had experience in the use of the apparatus, that a considerable proportion of the sulphur actually a component of the gas subjected to the examination escapes retention in the receiver, even when a second condenser is added. It will fall within my province, in the course of this paper, to show, by comparing the results obtained with the apparatus as set up under Dr. Letheby's guidance rules, without some improved methods, in the first place devised by M. Ellissen, and in the second place by myself, that the loss of sulphur from various imperfections may be fairly considered as enormous, and it is perfectly well established that this deficiency of so much of the sulphur is principally due to the uncertain and irregular combustion of the gas when the "Leslie" burner is employed to burn



it; for with the "single" jet a very steady and uniform flame may be easily maintained throughout the experiment. To the superiority of the "single" jet when substituted for the "Leslie" burner, as furnishing a marked increase in the quantity of sulphur in comparative experiments, we are entirely indebted to M. Ellissen, whose experiments are detailed in the report I have quoted above. I may observe here that all my own experiments, of which I have made a considerable number with every possible care, exhibit a very great loss of sulphur in each instance in which the "Leslie" burner is used instead of the "single" jet to burn the gas. However, the loss so incurred would be more or less compensated for by the increased proportion of sulphur condensed in the receivers, because of the atmospheric sulphurous acid, and possibly in some cases sulphuretted hydrogen, which could not fail to become absorbed in the receivers, especially when strong ammonia had been placed in these vessels. This addition of strong ammonia to the receiving condensers is a proceeding we must by no means overlook, unless we would incur the certainty of a loss of sulphur to an extent far greater than that which at all times, under the conditions described, is quite inevitable. We have a parallel in this case of compensation by increased accumulation of sulphur in the condensers from the sulphur of the air of towns, against loss of it by the defects mentioned as peculiar to the apparatus in the case of the celebrated arrangement of Liebig for the analysis of organic bodies.

Early on in the employment of this application it was found that the carbonic acid obtained through the combustion of the organic body submitted, mixed with the oxide of copper to analysis, uniformly afforded a proportion of carbonic acid lower than corresponded with the actual amount of carbon known to be present in the substance. But at the completion of the process of burning it was necessary to draw atmospheric air through the entire apparatus by an operation of suction, so that none of the carbonic acid remaining in it should avoid absorption by not being transmitted through solution of potassa placed in connexion for that purpose. It was remarked by those who used the apparatus at the outset, as a curious fact, that the small proportion of carbonic acid present in the atmosphere so drawn through increased the weight of the potassa apparatus to an extent generally closely coinciding with the proportion lost by the peculiar defect incidental to the process itself, and that it was as well, on that account, to neglect adopting a precaution never omitted by some chemists in such analyses—that of first connecting the one end of the apparatus with a tube of potassa before applying suction, so that the atmospheric air, before being drawn through, should be by that means entirely freed from the small quantity of carbonic acid which it always contains.

However this may be viewed as applicable to the question of sulphur determination in coal gas, it is quite certain that the adoption of a system which would conclusively obviate all chance of error of this description is extremely desirable.

My efforts to improve the apparatus as at present employed have led me to make a series of experiments with the view of ascertaining the possibility of burning the gas in an atmosphere where all

likelihood of contamination or augmentation of the condensed sulphurous products, by any sulphurous particles contained in that atmosphere, should be thoroughly prevented. Independently of the importance of securing this end, as enabling us to speak reliably and positively at any time regarding the maximum amount of bisulphide of carbon compounds in any certain specimen of gas, I need scarcely allude to the great convenience I have experienced, and which I feel sure others, as well, will find in the arrangement of apparatus I am presently going to describe. With it, sulphur determinations may be carried on in a laboratory where, from operations of other workers, sulphuretted hydrogen or sulphurous acid may be generated without the slightest possibility of absorption of either of these gases from the passage of the atmosphere of the laboratory containing them into and through the receivers of the apparatus.

I may now sum up the results of my experiments at the outset of the details belonging to them, and the conclusions I have arrived at under three heads:—First, a number of experiments have been made in which an exact comparison is shown of the results obtained by burning volumes of gas under the trumpet-shaped tube connected with the two receivers, containing uniform measures of strong ammonia, with the “single” jet and the “Leslie” burner.

Secondly—The results obtained by burning like volumes of another bulk of gas with the “Leslie” burner and Dr. Letheby’s arrangement, compared with the same gas burned with the “single” jet connected with my new apparatus, which I call the “Desulphurizer,” by the addition of which the atmosphere in which the gas is then burned is absolutely freed from all traces of sulphurous acid or sulphuretted hydrogen.

Thirdly—The final results, as I shall then demonstrate, of a large number of experiments comprised under the above first and second heads, are, that the apparatus used and recommended by Dr. Letheby, and the system of acidification likewise adopted by him, from constantly exhibiting in its action large losses of sulphur—*frequently exceeding 30 per cent.*—must be wholly abandoned for this purpose where anything approaching accuracy is sought for by the experimenter.

We must, if we would be accurate, definitively relinquish acidification and oxidation with nitric acid of the ammoniacal sulphite, and take care to employ instead nothing but chlorine for that purpose. There is imminent danger that the sulphate of barium, when precipitated by chloride of barium in the presence of much nitric acid, carries down with it an amount of nitrate of barium, which adheres to it with astonishing tenacity, notwithstanding copious washing with boiling distilled water be afterwards persevered with. Not only have we this source of error to contend with, but in cases where much ammonia has been placed in the receivers, and in consequence a considerable quantity of nitric acid has been added to produce sufficient acidification, a proportion of nitrate of ammonia would then be present in the solution from which we have subsequently to precipitate by the addition of the baryta salt, that must, as I have found, interfere in such a way with the precipitation of the sulphate of barium as to destroy



all confidence in the issue of the experiment. To prove how completely all reliability must be misplaced, when reposed upon any experiment conducted upon the nitric acid acidification principle, I must quote the most eminent quantitative analyst of the day, M. Remigius Fresenius, who observes as follows, at page 104 of his last (fourth) edition of the "System of Quantitative Analysis," published recently:—"Sulphate of barium, when recently precipitated, it is difficult to obtain a clear filtrate from, especially *if the precipitation was effected without the aid of heat*, and the solution contains neither hydrochloric acid, nor chloride of ammonium. It is insoluble in cold and in hot water. It has a great tendency upon precipitation to carry down with it other substances contained in the solution from which it separates, more particularly nitrate of barium, chloride of barium, &c. These substances can generally be completely removed only after ignition by washing with appropriate solvents. Even the precipitate obtained from a solution of chloride of barium by means of sulphuric acid in excess contains traces of chloride of barium which it is impossible to remove, even by washing with boiling water, but which are dissolved by nitric acid. Cold dilute acid dissolves trifling yet appreciable traces of sulphate of barium: for instance, 1000 parts of nitric acid, of specific gravity of 1.032, dissolve 0.062 parts of sulphate of barium. Cold concentrated acids dissolve considerably more: thus 1000 parts of nitric acid, of 1.167 specific gravity, dissolve 2 parts of sulphate of barium. Boiling hydrochloric acid also dissolves appreciable traces. Acetic acid dissolves less sulphate of barium than other acids. Free chlorine considerably increases the solubility of sulphate of barium. Several salts more particularly interfere with the precipitation of sulphate of barium. I observed this some time ago with chloride of magnesium, but *nitrate of ammonium possesses this property in a high degree.*" Again, page 266, in speaking of the precipitation of sulphate of barium by the addition of chloride of barium to the solution which must be acidified by hydrochloric acid, to acid reaction, he says: "Should the analyzed solution contain nitric acid, some nitrate of barium is likely to precipitate in conjunction with the sulphate of barium: the removal of this admixture of nitrate of barium from the precipitate requires protracted washing with hot water. It is, under all circumstances, necessary to continue the washing of the precipitate until the last washings remain perfectly clear upon testing with sulphuric acid. In cases where perfect accuracy is desirable, I would recommend the following proceeding:—After igniting the precipitate, and weighing, moisten it with a few drops of hydrochloric acid; add hot water, stir with a very thin glass rod, rinse the rod, and warm gently for some time. Pour the almost clear fluid upon a small filter paper in a glass funnel, and test the filtered fluid with sulphuric acid. If this produces turbidity or a precipitate, which is a sign that the sulphate contains an admixture of another barium salt, wash the residue (the sulphate) again with hot water, until the washings are no longer rendered turbid by sulphuric acid. Dry now the precipitate, ignite, and weigh. If sulphuric acid has been precipitated from a solution containing much nitric acid or much alkaline salt, the testing of the ignited precipitate is not merely to be recommended, but is absolutely necessary, since in such case it is by no

means unlikely that the sulphate of barium will contain 1 per cent. or more of nitrate of barium or alkaline salt. *The results are not always so exact as used to be believed.* If precipitated in very acid solutions, a little of the sulphate of barium remains dissolved. If precipitated in very saline solutions, on the other hand, the results are generally too high, since it is difficult in this case to obtain a pure precipitate of sulphate of barium. The sulphate of barium has a great tendency to carry salts (especially nitrates and chlorides) down with it which cannot be removed at all by washing, and are removed but imperfectly often when the ignited precipitate is treated (as above described) with hydrochloric acid and water."

Now, it can be but folly in any analyst who would continue to employ nitric acid as the oxidizing agent with the certainty of sustaining a loss from the contingencies so clearly demonstrated in the above extracts from the work of M. Fresenius. And the small bulk of gas generally at our disposal to be submitted to the burning operation, from furnishing but a comparatively minute quantity of barytic sulphate, obviously makes the chance of error more serious—on the one hand from loss by non-precipitation of some of the sulphate of barium, owing to the presence of much nitrate of ammonium; or, on the other hand, from nitrate of barium adhering obstinately to the sulphate of barium; from which, as we see from Fresenius' experiments, and as I have myself experienced, it is very often quite out of our power to remove it.

It is to this cause that such startling discrepancies in results are so frequently found in many experiments.

In my papers published last year in this JOURNAL, I gave some outline of the way I proceed to estimate the sulphur. I need hardly say that I still employ nothing but a current of chlorine for the oxidation of the sulphite of ammonium into sulphate. It entails no more inconvenience than with the use of nitric acid; and and if it did, if we desire anything approaching accuracy, we must nevertheless continue its use. We can simply and easily effect the desired end on the following plan:—When the contents of the two condensing vessels are drained out, and thoroughly washed clean with pretty hot distilled water, they may be mixed in a capacious vessel with the washings of the cork junctions and the trumpet-shaped tube. All parts of the apparatus with which the sulphurous ammoniacal fumes have come in contact should be repeatedly washed with hot distilled water, until they fail to produce the *slightest* turbidity with dilute solution of chloride of barium after standing at least a quarter of an hour. The mixed solutions must then be cautiously concentrated by gentle boiling in a capacious evaporating-dish. It is necessary to be extremely cautious how we apply the necessary heat, for if we neglect to watch the heating operation, an exceedingly brisk effervescence, resulting from a rapid evolution of carbonic acid from the decomposition by the elevated temperature of carbonates of ammonium, into which the strong ammonia of the receiver has become converted, would cause some degree of loss through particles of the fluid becoming projected over the sides of dish. When the evaporation has been carried so far that only 2 or 3 fluid ounces of solution remain, a beaker-glass is selected in which it is proposed to mix the fluid, when properly treated, with the chloride of barium for subsequent



precipitation. Then the solution is carefully poured from the evaporating-dish down a stirring-rod into this beaker-glass; and then what fluid remains adhering to the evaporating-dish is cautiously also transferred into it by one or two rinsings with hot water from a laboratory washing-bottle of the most convenient form.

We need not concern ourselves at this stage to transfer every particle of adherent solution of the dish into the beaker-glass when we have twice washed it out with hot water; for the contents of the beaker-glass being then forthwith treated, while occasionally stirred with a spatula, with a current of chlorine emitted from a glass tube brought as closely as possible to the bottom of the vessel, until the green gas is seen to escape unabsorbed, and form an atmosphere above the liquid, are shortly afterwards again put back into the evaporating-dish. The chlorine should be evolved in a little flask from about half an ounce of good black oxide of manganese, and a little more than sufficient pure hydrochloric acid to cover it, a gentle heat being cautiously applied. An intermediate washing-bottle of hot water must be employed to wash the gas; and the junctions of the tubes may be made with vulcanized india-rubber tubes, which, however, must at least have been used two or three times previously, and where chlorine has been in contact with their inner surfaces. When the beaker-glass and the tube through which the chlorine has been passed into the solution have been rinsed clean and added to the contents of the dish, these are once more concentrated, with careful gentle boiling to expel the chlorine, to the bulk of an ounce or two; and, as the solution usually contains fragments of cork, it is then filtered through as small a paper filter as can be selected for convenient use. The beaker receives the filtered fluid.\* Three additions of hot water to the filter after it has drained thorough I uniformly find suffices to free it from all adhering sulphate. We may assure ourselves of this by allowing the fourth addition of hot water from our "wash" bottle to run into a small beaker, into which we have poured the small quantity of chloride of barium intended to be subsequently thrown into the treated solution for the subsequent precipitation. I always find that every trace is so absolutely removed at that stage, that not a particle of sulphuric acid can be found in the final fourth drainings off.

One of the several advantages resulting from the use of chlorine instead of nitric acid is, that with the first we always obtain, after concentration by boiling (to get rid of any free chlorine—a step of the highest importance) and filtration, a perfectly clean limpid fluid, from which, by the addition of the requisite quantity of the barytic precipitant, a precipitate is instantly obtained, which, after heating nearly to boiling for a few minutes, subsides rapidly. Yet we must on no account attempt to collect it on a filter until it has stood at least twelve hours. It never passes through the filter if we provide ourselves with good filtering paper. On the contrary, when we employ nitric acid, we frequently obtain a dirty viscid brown, somewhat turbid, solution. Here we miss the destructive,

\* The dish is best brought thoroughly clean by means of hot water and a clean feather on the end of a quill.

bleaching action of the chlorine upon organic matters, which always accompany the salts removed from the receivers, &c. That these organic matters, where nitric acid is used, likewise retard the precipitation of the sulphate of baryta, I feel very certain, and that they, together with the nitrate of ammonia, prevent the deposition of some portions of it entirely I know quite well.\*

I enter thus very completely into a description of the best mode of proceeding as regards these particular manipulations in estimations of coal gas sulphur, which I always follow, because of my having received from various sources numerous applications upon the subject since the few details I gave thereon in the series of articles published in this JOURNAL last year upon the "Action of the Sulphides of Ammonium upon the Bisulphide of Carbon and Sulphuretted Hydrocarbons in Coal Gas."

Regarding the comparative results obtained in the determination of the proportion of sulphur in the form of bisulphide of carbon or sulphuretted hydrocarbons in a volume of coking coal gas divided in two equal bulks—the first bulk being burned by the "Leslie" burner, and the second consumed by a "single" jet—

*Experiment A.*—The first half of the volume of gas taken for this estimation was transferred to the gasholder F, figured at pages 569 and 658 of vol. xiv. of this JOURNAL. All in excess of  $2\frac{1}{2}$  feet exactly was let off by the surface stop-cock. Rather more than 5 cubic feet had been collected in the largest gasholder, A (p. 569, vol. xiv.), a few days previously, and allowed to stand over water, the level of which was during that period preserved at *e' e*. An absorption of gas is generally remarked by such contact of coal gas with a surface of water in a vessel of this description. There is a curious degree of variation in this amount of absorption, however, I have observed, with bulks of gas taken at different times from a company's supply, notwithstanding that the *illuminating power throughout may be extremely uniform in value*. When no more absorption was observed, the  $2\frac{1}{2}$  feet was forced from A, through the stop-cock C, connected by a long tube with stop-cock *f*, of the gasometer F, by a head of water joined from the tap H' to the inlet of large holder A into gasholder F, for burning off.

Six hours were occupied by the consumption of this first  $2\frac{1}{2}$  cubic feet, and an ample supply of strong ammonia was placed in the bottle underneath the funnel supporting the "Leslie" burner. Two receivers were placed to collect the products of the combustion, each containing two fluid ounces of the strongest solution of ammonia absolutely free from any traces of sulphuric acid. To have conducted the experiment with greater rapidity would have been to sacrifice the advantage desired in this case of maintaining the steadiest flame attainable with this kind of jet, so as to make sure of the highest possible result in the sulphur obtained.

Experiment 1 had been commenced so early in the day that immediately the burning off was concluded the remaining half of the gas in A should be immediately passed into the gasometer F, to be

\* I always experience in precipitating  $\text{SO}_3\text{BaO}$ , after treating as described by chlorine, that we have the solution acidified to a uniform degree in all experiments, which cannot be attained by any other means.

in turn, with as little delay as possible,\* measured and burned by the "single" jet.

*Experiment B.*—To this end, a second set of receivers and another trumpet tube, &c., were provided, but of precisely similar size and construction, so that the whole apparatus should be set up and set going without delay. Two fluid ounces of strong ammonia from the same stock as that used in the preceding experiment were added, as before, to each receiver, and a quantity of strong ammonia similar in bulk to that employed under the funnel in last experiment was placed under the funnel in this. The combustion lasted six hours. In a word, every detail was observed exactly as in experiment 1, with the one exception that the "single" jet burner was used in experiment 2.

The following are the results of these two experiments:—

*Table showing Results obtained by burning two equal measured bulks of Coal Gas, one with a "Leslie" Burner, and another with a "Single" Jet; all other conditions in the arrangement of the apparatus (including oxidation with chlorine) being in both experiments precisely similar.*

Experiments.	Quantity of Gas analyzed.	Description of Burner.	Weight of Sulphate of Barium obtained.	Amount of Sulphur calculated for 100 Cubic Feet.
A ..	2½ c. feet	..... Leslie	1.56	8.56
B ..	2½ c. feet	..... Single jet	2.17	11.92

Note.—The difference between 11.92 and 8.56 is 3.36, which shows a loss of 39.29 per cent. of sulphur through using the "Leslie" burner instead of the "single" jet.

In the next two experiments a grain or two of bisulphide of carbon were swept into the largest gasholder by the current of gas at the time of filling it from the company's supply. It was then allowed to stand as before, over water for one week, to allow time for thorough diffusion. Two quantities of 2½ feet respectively were then very carefully burned with the same precautions taken in the two last experiments.

*Table showing Results obtained by burning two equal measured bulks of Coal Gas, into which a little Bisulphide of Carbon Vapour had been diffused; one portion being consumed with the "Leslie" Burner, the second part being burned with the "Single" Jet.*

Experiments.	Quantity of Gas analyzed.	Description of Burner.	Weight of Sulphate of Barium obtained.	Amount of Sulphur calculated for 100 Cubic Feet.
C ..	2½ c. feet	..... Leslie	2.61	14.33 gr.
D ..	2½ c. feet	..... Single jet	4.41	24.22 gr.

Then 14.33 : 9.89 :: 100 : 69.01 per cent. loss through employing the "Leslie" burner.

The whole of the succeeding experiments further made to clearly establish the loss incidental to the use of the "Leslie" burner, are placed in the following table.

\* I have repeatedly satisfied myself that after gas had been allowed to stand a day or two over water in my gasholders, as described, no appreciable alteration of nature occurred to it by remaining there for another day. However, for all this, I never omitted burning the two bulks of gas in one day.



Table showing Results of burning two equal bulks of Coal Gas, with, in the one case, the "Leslie" Burner, in the second, the "Single" Jet. About six hours were required for the duration of burning in each experiment.

Experiments.	Quantity of Gas analyzed.	Description of Burner used.	Weight of Sulphate of Barium obtained.	Quantity of Sulphur calculated for 100 Cubic Feet.	Loss of Sulphur per cent. through using the Leslie Jet instead of the Single one.
E ..	2½ c. feet ..	Leslie ..	1.90 ..	10.40	} 16.15 gr.
F ..	2½ c. feet ..	Single jet ..	2.20 ..	12.08	
G ..	2½ c. feet ..	Leslie ..	1.60 ..	8.76	} 44.74 gr.
H ..	2½ c. feet ..	Single jet ..	2.31 ..	12.68	
I ..	2½ c. feet ..	Leslie ..	1.32 ..	7.24	} 97.23 gr.
K ..	2½ c. feet ..	Single jet ..	2.60 ..	14.28	
L ..	2½ c. feet ..	Leslie ..	2.00 ..	10.96	} 30.65 gr.
M ..	2½ c. feet ..	Single jet ..	2.61 ..	14.32	
N ..	2½ c. feet ..	Leslie ..	1.82 ..	9.96	} 21.28 gr.
O ..	2½ c. feet ..	Single jet ..	2.20 ..	12.08	
P ..	2½ c. feet ..	Leslie ..	2.20 ..	12.08	} 23.17 gr.
Q ..	2½ c. feet ..	Single jet ..	2.71 ..	14.88	
R ..	2½ c. feet ..	Leslie ..	1.87 ..	10.24	} 21.09 gr.
S ..	2½ c. feet ..	Single jet ..	2.26 ..	12.40	
T ..	2½ c. feet ..	Leslie ..	1.61 ..	8.84	} 48.41 gr.
U ..	2½ c. feet ..	Single jet ..	2.39 ..	13.12	

By adding up the results of the eight experiments in the above table, made with the "Leslie" burner, we obtain the numbers 14.32, which divided by 8 gives the number 1.79 as the mean of the whole. Similarly, the sum total of the other eight combustions gives the figures 19.28, which also divided by 8 leaves 2.41 for the "single" jet mean. Proceeding in the same way with the fourth column of the table—the quantity of sulphur calculated for 100 cubic feet—we get the average 9.81 grains of the element for the burnings by the "Leslie" burner, and 13.23 grains for those in which the "single" jet was used. If we add together the eight quantities giving the loss of sulphur incurred through the employment of the "Leslie" burner, we have, as the average of this, 37.84 per cent. We obtain, however, the exact per centage of loss through substituting the "Leslie" burner for the "single" jet in the sixteen experiments, as a mean result, by the following calculation:—

	In 2½ feet.	In 100 feet.
Mean of 8 experiments, "single" jet	2.41	13.23
Mean of 8 experiments, "Leslie" burner	1.79	9.81

Loss by using the "Leslie" .... .62 .... 3.42

Then 1.79 : .62 :: 100 : 34.63 for 2½ feet of gas.

And 9.81 : 3.42 :: 100 : 34.86 for 100 do. do.

I extract and annex underneath, from the report of M. Ellissen (JOURNAL OF GAS LIGHTING, Oct. 18, 1864, vol. xiii., p. 717), the results of the six experiments made by him to compare the quantities of sulphur determined in a bulk of gas by the "single" jet and "Leslie" burner.

	By "Leslie" Burner.	By "Single" Jet.
1st experiment....	4.31 .....	6.88
2nd do. ....	5.09 .....	4.96
3rd do. ....	3.44 .....	4.05
Mean ....	1.42 .....	1.76

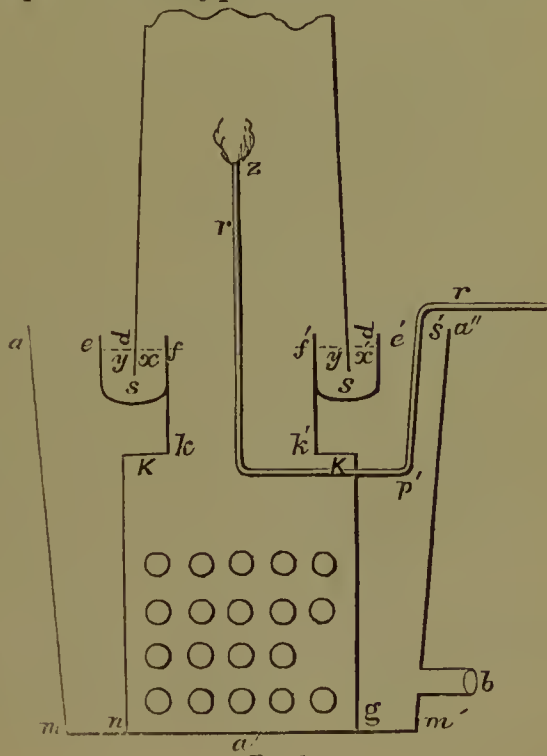
Then the proportion,

$$1.42 : .339 :: 100 : 23.76,$$

shows that slightly above *twenty-three and three-quarters* per cent. of sulphur was lost because the "Leslie" burner was employed instead of the "single" one!

If we take the means of M. Ellissen's first and third experiments, we find that in the place of the above loss, we have a deficiency instead of 41 per cent. in round numbers from the same cause! For the difference between 5.46 (the mean of the "single" jet determinations) and 3.87 (that of the "Leslie" estimation) is 1.59, and  $3.87 : 1.59 :: 100 : 41.09$ . I consider this average to be much nearer the truth—first, because I never yet obtained otherwise, *at the lowest, than from 16 to 20 per cent. of loss* (as compared with the "single" jet) by using the "Leslie" burner; and secondly, because it so nearly corroborates the considerable number of experiments given in the above tables.

*Of the Method of Burning the Gas with the addition of an Apparatus I call a "Desulphurizer," by which any Increase of Sulphur in the Condensed Products from the surrounding Atmosphere is entirely prevented.*



I must now refer to the annexed drawings. Fig. 1— $a a' a''$  is a vessel made of sheet iron, with an outlet of half an inch in diameter, capable of being closed by a cork, a little above the bottom at  $b$ . This vessel is circular;  $5\frac{1}{2}$  inches deep, 6 inches across the top (from  $a$  to  $a''$ ), diminishing gradually in diameter towards the bottom, where its measurement is only 5 inches.

Fig. 2 shows the interior part of the apparatus, which we must imagine has been lifted out—for it may of course be removed if necessary—of the external vessel  $a a' a''$ . It consists of another cylindrical formed vessel  $K n K g'$  of sheet iron, open at the top and bottom at  $ff'$  and  $n g'$ . It measures  $3\frac{1}{2}$  inches in diameter and 4 inches from the lowest part,  $n$ , to the upper surface,  $K$ . All around the lowest part of this inside cylinder, as high as 3 inches up, but no further, it is punched full of holes, similar to a vegetable colander, of about, or somewhat less, than half an inch

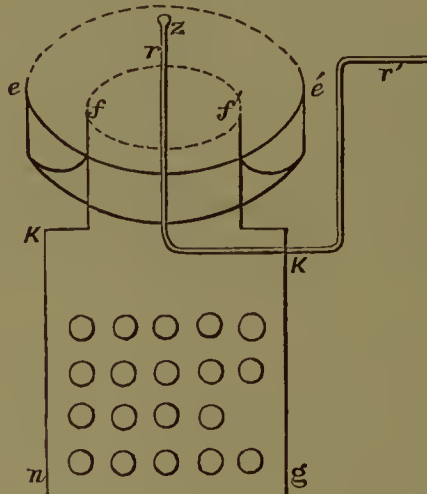


FIG 2.

in diameter. It is turned in horizontally, and vertically again, as shown in Fig. 1 at  $K K$ , where it is narrowed at  $k k' f f'$  to a diameter of 2 inches across. This contracted circular mouth (as I may express it) is cemented perfectly air-tight in a hollow circular glass gutter shown at  $e f f' e'$ . This glass gutter is 4 inches across from  $e$  to  $e'$ , Figs. 1 and 2, 1 inch wide from  $e$  to  $f$ , and 1 inch in depth from  $p$  to  $s$ . The office fulfilled by it is to act, when filled with a fluid, as a close joint, when the lower end of the trumpet-shaped tube,  $o$ , is lowered carefully into it; for as it is, besides being 1 inch in width all round, 1 inch in depth, as these trumpet-shaped tubes are generally made, no difficulty is, I always find, experienced in placing the tube well into the positions shown at  $x x'$  in the drawing;  $r r$  is the metal pipe, with a single jet,  $z$ , attached, through which the gas is conveyed as usual to the inside of the trumpet-tube—projecting up it about 3 inches—for burning. This pipe is soldered into the side of the inner perforated vessel,  $n g' K K$ , and passes up through the circular orifice (which is of the diameter of 2 inches),  $f f'$ , to its jet,  $z$ .



Having described the construction of the apparatus, I will now give an account of the mode in which it is employed to effect the desired end—that of enabling us to determine the sulphur in coal gas, no matter how sulphurous the external atmosphere may be at the time of the experiment:—

The gas being in complete readiness for burning, the interior perforated cylinder,  $K n K g$ , is placed inside the external vessel,  $a a' m m'$ . The receivers being charged with the proper quantity of strong liquid ammonia, are then joined to the trumpet-tube, and raised to such an elevation upon the table, that the vessels, with their gas-supply tube,  $r r$ , may be easily placed in their proper position under the trumpet-tube at any moment. Upon looking at the arrangement made, as sketched in Fig. 2, it will be seen that between the holes of the inside cylinder  $K n g$  and the inner walls of the outside one,  $a a' a''$ , there is a considerable space open, increasing from about three-quarters of an inch at  $g$  and  $n$  to about  $1\frac{1}{4}$  inch at the part  $p'$ , and the same for the corresponding place on the opposite left-hand side and all round the vessel. Every part of this vacancy around and over the holes of the inside cylinder is then filled in and covered to within about an inch of the top of the inside of the exterior vessel with fragments of pumice-stone the size of very small nuts, which have been just before thoroughly soaked in a moderately strong solution of potassa. Lastly, as much strong ammonia is then to be poured into the glass gutter,  $e f f' e'$ , as will, when the trumpet-tube is depressed below the surface of the ammonia in the gutter (shown at the dotted level line at  $y y'$ ), by raising the whole arrangement upon blocks until the proper height is attained, suffice to form a completely close ammonia joint, and so prevent any air entering except that which passes, first down into that part of the apparatus  $e e s'$  (Fig. 1), where indicated by the arrows. Thence, in its passage to the interior of the perforated inside, into which a current is instantly established when the gas is lighted, it comes very thoroughly in contact with large surfaces of potassa solution soaked into the pieces of pumice. When it passes upwards as a current of hot air circulates up the trumpet-tube in the direction further shown by the arrows, finally emerging by the orifice  $f f'$ , on its road to burn the gas at the jet  $z$ , it has been by that time completely deprived of all sulphurous particles which are capable of absorption by potassa solution.

It is a manipulation of the greatest simplicity when the receivers and trumpet-tube are placed at the required level; and it has been further ascertained, by a trial just previously, how much ammonia need be poured into the gutter-lute, to light the jet, and place it beneath the trumpet-tube, out of contact with any but the purified atmosphere, and so commence the combustion of the gas at any desired moment.

I propose in future pages of this work to give an account of numerous curiously interesting results obtained by comparing the amounts of sulphur determined with this additional apparatus, with those obtained by employing both the "Leslie" burner and "single" jet as given above.

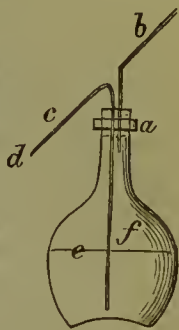
One effect produced from the air being thus caused to circulate through the interstices of the pieces of pumice in this "Desulphu-

rizer" before being admitted in contact with the burning jet  $z$  in the interior of the trumpet-tube, is, that the enclosed atmosphere is maintained throughout the experiment in a very still condition. The atmospheric air drawn in to replace the heated portions successively passing up the trumpet-tube into the condensers flows in continuously with complete regularity. The frequently remarked flickering of flame which will sometimes happen with the best description of single jet employed to burn the gas under the trumpet-tube when open to the external air, not unfrequently arising from such causes as the sudden opening or closing of a door or window, or the rapid movement of any one to and fro in the room, is, by its use, entirely prevented. In fact, we have in this arrangement the nearest possible approach, I think, to a great desideratum; the combustion of the gas in a vessel so far closed as to be entirely free from such external disturbing sources of inaccuracy. In all other construction of apparatus of this kind hitherto described to accomplish this purpose, as I have now shown, we have had to contend with loss of sulphur from imperfect burning of the gas, and error from probable condensed sulphurous component parts of the atmosphere becoming retained in the receiving vessels. We shall see presently that the combustion of the gas in a quiet flowing current of air, controlled in its influx by its passage through the small lumps of pumice, affords results which, while almost wholly unanticipated by myself at the outset of these experiments, can only be explained upon the ground that when the gas is so burned its combustion is, if not quite, very nearly perfect. From the fact that, by the use of this additional pumice apparatus, the ascending column of air, heated inside the trumpet-tube by the jet of gas burning there, becomes so much reduced in velocity, I feared the possibility of some portions of the sulphurous fumes produced about, or a little above the burning jet  $z$  descending and subsequently getting absorbed, not only by the ammonia in the glass gutter  $e f f' e'$ , but also by the potassa soaked into the pumice lying against the perforated cylinder  $K n K g$ . Therefore, I have given particular attention to ascertaining exactly whether any source of error of this kind is incidental to the employment of this "Desulphurizer." When each experiment, of which I am presently going to give the full details, in which it had been put in action had terminated, the gutter-lute was emptied of its contents. To effect this, I may mention there is no necessity to remove the interior cylinder from the outer one. To do so after making each experiment would be a troublesome, and, owing to the use of potassa solution, a disagreeable task. Every trace of ammoniacal liquor remaining in the "gutter" can be readily removed with a small right-angle shaped glass syphon of one-eighth of an inch internal diameter in the following manner:—The syphon is filled with distilled water, and the shortest limb, in which the water is sustained by the pressure of the atmosphere when it is inverted by previously placing the finger over the other opening, is introduced beneath the fluid in the "gutter." Nearly every drop may be thus run off from it into any suitable vessel placed to receive it underneath the longest limb of the syphon by cautiously tilting it upon one side as its contents are removed; after which the syphon should be washed



clean by the best adapted wash-bottle which can be obtained for this purpose. I have so often had occasion to refer to the employment of this washing-bottle, that it may be useful to many working at this subject, who are not entirely devoted to chemical pursuits, if I at once describe that which I prefer.

The washing-bottle best adapted for these manipulations is that figured upon the margin. A good soft cork (it is not quite safe to use india-rubber vulcanite ones where sulphur determinations are the subject of experiment, otherwise they are very convenient) is fitted to the mouth of the flask at *a*, *d* which has two holes neatly perforated in it, through which are passed the two glass tubes, *b* and *c*. It will be seen that the tube *c* passes nearly to the bottom of the flask, and beneath the surface of the water at *e*. The tube *b* merely passes through the cork. The tube *c* is drawn out to a tolerably fine orifice at *d*. When the air occupying the space above the water *f* is compressed by blowing with the mouth down the tube *b*, the water *e* is propelled by the pressure up the tube *c*, and is then forced in a fine stream from the opening *d*, with an intensity varying with the force with which air is driven by the lungs into the tube *b*. When the flask is inverted a stream or pencil of water flows with a convenient rapidity from the tube *b*, which is thus supplied in a much more speedy manner than when blown from the smaller outlet *d*. Both of these jets of water are invaluable for rinsing the receivers, tubes, &c., to which I have made allusion in my papers; and if warm or hot water is wanted for such purposes, it is a matter of great simplicity to keep the flask (or a second stock one from which it may be replenished) standing upon a tripod over a suitable gas-flame until the water it contains has been raised by the heat to the required temperature.



Now, when the glass gutter has been rinsed by blowing warm water from the smaller orifice of this bottle upon its interior, and running it off twice by the cleaned syphon, it is always in my experience, quite clean, and ready to be charged with the ammonia to be employed as the seal in the succeeding experiment. It was in this way that I assured myself, at the conclusion of every experiment that I have hitherto made, of the certainty that no sulphurous compounds whatever become absorbed there whilst a combustion operation is in progress. That is to say, I removed repeatedly the liquor from the glass gutter at the end of the process, passed chlorine through it, boiled, and mixed it with a little chloride of barium. As a result of every trial, I never obtained the slightest turbidity, proving the entire freedom of the liquid from any trace of sulphur compounds.\* This, then, being the case, it may with complete certainty be deemed as quite conclusive that no sulphurous particles can get absorbed by the potassa soaked pumice through their descending (or diffusing) down the contracted opening *ff'* into the

\* A few days ago, having occasion to estimate the sulphur in the apparatus as drawn by Dr. Letheby for the *Chemical News*, after 5 feet of gas had been burned, I tested the ammonia of the bottle placed under the funnel and Leslie burner for sulphuric acid in a like manner. I did not find a trace there.



perforated chamber *K K n g*, and so coming into contact with these packed outside it.

It is a safe precaution, however, never to omit adding the contents (and washings) of the "gutter" to the products of the gas burning of the receivers, &c., when an experiment is finished. If this be not done, they must be separately tested, as I have described, for sulphuric acid. All this is necessary for the following reasons:—An operator not sufficiently accustomed to the use of the pumice apparatus and the "single" jet, might possibly burn the gas with too small a flame. This would cause some part of the products of combustion, instead of being wholly carried into the condensers, to attach themselves to the sides of the trumpet-tube, because of its not being kept hot enough, which is sometimes the case when  $2\frac{1}{2}$  cubic feet occupy more than six hours to burn. If we do not require more than  $5\frac{1}{2}$  hours time to consume  $2\frac{1}{2}$  cubic feet, we avoid this condensation inside the trumpet-tube. This is of no importance whatever when the trumpet-tube dips into the gutter, and we take care to subsequently add its contents to the other products, for the drops of fluid, as they trickle down the first, run into and are received by the last. But if there be no gutter placed under the trumpet-tube, as in the open arrangement, then it is not an unfrequent occurrence for these drops to fall upon the table and become a loss in the analysis. Of course it is an important loss from the fact that drops of fluid in flowing down the interior of the upright tube wash down no inconsiderable portion of sulphite and sulphate of ammonium, which always collects there in notable quantity.\*

When the pumice apparatus has been used so many times that it is considered necessary to again soak the fragments of pumice in fresh potash, we may further save ourselves some loss of time and a little unnecessary trouble by proceeding as follows:—It will be recollected that an outlet has been described as provided near the bottom of the outside vessel, in the inside of which these pieces of pumice are placed. It is shown in the drawing at page 617, at *b*. Now when the spaces *a K n m* and *a "K g m,"* which extend around the inside perforated cylinder and the outer one, are filled with the pumice fragments, the solution of potassa prepared for the soaking process is to be frequently poured over these, so as to bring it into contact as much as can be with every piece of pumice in the vessel. After the solution has drained to the bottom, it is poured out from the opening *b*, and when this has been corked tightly it is again thrown with care upon the pumice: drawn off, and the same procedure repeated until it is quite certain all the pumice is well saturated by the potassa. It is important that the utmost caution be exercised lest, in putting the potassa solution over the pumice, some of it is by chance conveyed into the glass gutter. If any by an accident of this kind fell there, as the potassa used in this case would undoubtedly contain a considerable quantity of sulphuric acid, it is evident a serious cause of error would occur in the next analysis in which the gutter would again be used.

The experiments made to compare the results obtained by burn-

\* See page 659, vol. xiv.

ing precisely similar volumes of a selected bulk of gas, first, with either the "single" or the "Leslie" burner, over ammonia, with free access of common air, and secondly, with the jets enclosed by the "Desulphurizer," are now to be described. It should be mentioned, however, in the first place, that the samples of gas taken in each case were stored in the gasholders A and B (p. 569, vol. xiv.) in the following manner:—The inlet D was closely plugged by a good cork. The tap C was then opened, and water from the tap H' was conveyed by a flexible tube to it until it overflowed there. Then the tap C was shut off, and joined to a supply stop-cock of the company's main. When the cork by which the opening D had been closed was removed, after that opening had been covered with water by pouring it into the small cistern E, the gasholder A remained filled with water supported by the pressure of the atmosphere. When, again, the tap C and the stop-cock of the company's main were cautiously partially opened, all the water which ran away through the opening *e* was replaced by gas. In this way gasholders may be very conveniently and expeditiously filled with gas from a company's main.

The first experiments of comparison were made with the contents of gasholder A filled from the company's main as above described;  $2\frac{1}{2}$  feet were transferred from it to the gasometer F (vol. xiv., pp. 569 and 658), and burned with the same "single" jet used in all the experiments of comparison with the "Leslie" jet detailed at page 616, vol. xv. The same apparatus, indeed, was employed in this case as in those instances. The second equal bulk of this gas was, in turn, burned inside the "Desulphurizer" on the afternoon of the same day, so soon as the apparatus used in the first combustion could be washed clean and set up ready for the experiment to be commenced. It had been previously arranged, by taking care that the "single" jet, which, as I have above observed, has been constantly in requisition in all my experiments, should be capable of transfer at any time from above the funnel figured at page 658, vol. xiv., to the inside upright pipe of the "Desulphurizer," page 617, vol. xv. As the connecting-screw threads of both of these vessels were alike, it could be placed in connexion with either in a few moments.

In the following table the results of these two combustions are appended:—

*Table showing the Results obtained by burning two equal measured bulks of Coal Gas. They were both burned with the same Jet, but in one case the new apparatus, called the "Desulphurizer," was used in addition. All other conditions except this (including oxidation by chlorine and employment of the same vessels) were precisely similar.*

Experiments.	Quantity of Gas analyzed.	Description of Burner used.	Whether "Desulphurizer" was used or not.	Weight of Sulphate of Barium obtained.	Amount of Sulphur calculated for 100 Cubic Feet.
V	$2\frac{1}{2}$ c. feet	Single jet	No	1.61 gr.	8.84 gr.
W	$2\frac{1}{2}$ c. feet	Same jet	Yes	2.21 gr.	12.12 gr.

Then the difference between the numbers obtained by the use of the "single" jet, 8.84, and those furnished by the addition of the "Desulphurizer," 12.12, is 3.28; and

$$8.84 : 3.28 :: 100 : 37.1 \text{ per cent.}$$



From these first experiments of comparison, it appears that more than one-third of sulphur increase is capable of determination by the addition of the "Desulphurizer" apparatus. This result was so unlooked for, that a number of other experiments in confirmation were forthwith put in hand.

Both the gasholders A and B were (vol. xiv., p. 569) for this purpose simultaneously filled with gas from the company's main. Three experiments (following in immediate succession) were immediately commenced. The results are tabulated underneath:—

*Table showing the Results obtained by burning three equal measured bulks of Coal Gas. All three were burned with the same Jet, but in two instances the new apparatus, called the "Desulphurizer," was employed in addition. All other conditions except this (including oxidation by chlorine, the use of the same condensers, &c.) were precisely similar.*

Experiments.	Quantity of Gas analyzed.	Description of Burner used.	Whether "Desulphurizer" was used or not.	Weight of Sulphate of Barium obtained.	Quantity of Sulphur calculated for 100 Cubic Feet.
X	2½ o. feet ..	Single jet ..	No ..	2.10 gr. ..	11.52 gr.
Y	2½ e. feet ..	Same jet ..	Yes ..	2.69 gr. ..	14.76 gr.
Z	2½ e. feet ..	Same jet ..	Yes ..	2.70 gr. ..	14.80 gr.

The mean of the last two experiments are the numbers 14.78 grains. The difference between these, and the numbers obtained without the addition of the "Desulphurizer," is 3.26 grains. Then—

$$11.52 : 3.26 :: 100 : 28.3$$

which calculation demonstrates that a loss of sulphur to the extent of *twenty-eight and one-third per cent.* arose through employing the "single" jet to burn the gas without applying that jet for that purpose in conjunction with the "Desulphurizer."

There is such a close coincidence in the amounts of sulphate of barium obtained in the experiments Y and Z—which it will be seen by a reference to the table are the two made in which the "Desulphurizer" was brought into requisition—that one might be almost inclined to consider that by such system of proceeding we have at length arrived at a method by which the whole of the sulphur existing in a bulk of coal gas would be susceptible of exact determination. More than a year ago I found that a great degree of uniformity attended determinations by the "single" jet, simply, as generally done, placed over the bottle of ammonia, provided all the conditions under which the different experiments were made were preserved in close uniformity. A reference to some experiments of mine made in August, 1865, and published at page 658, vol. xiv., demonstrates this in a singular manner. Four experiments made at that time to ascertain the degree of accuracy attainable by using the "single" jet to burn a volume of gas divided into four equal bulks gave, as there recorded, the respective quantities of sulphur at 13.11, 12.51, 12.34, and 12.64, each being calculated to correspond to 100 cubic feet of gas. As we now know it to be conclusively established that it is quite impossible to determine *all* the sulphur in a coal gas by using the "single" jet to burn it in the manner there described, notwithstanding the very close agreement obtained in the four experiments given above, it must not be assumed for one moment either that the close approximation between the numbers 14.76 and 14.80



given by the pumice apparatus addition any further contribute towards proving this possibility.

It is a curious fact, however, which may be deduced as a perfectly safe inference from the results of these six experiments, assuming, as most probable, that in not one of them did I succeed in arresting the *whole* of the sulphur, that the quantity of sulphur lost in these cases, provided the conditions under which the experiments are conducted possess very great uniformity, is proportionate to an *almost exact degree*. I must confess, however, that it is anything but easy to understand how this should necessarily always happen in a coincident rate in several analyses, when methods corresponding to an average manner of proceeding are followed, in *all* of which we cannot fail to ensure some loss from defects inseparable from the system. Are there *some* sulphur constituents present in coal gas which resist with great obstinacy, by even slow yet very complete burning in atmospheric air, oxidation into sulphurous acid? I have records of experiments in which "Leslie's" burner was resorted to for the estimation of the sulphur in the usual manner in a bulk of gas divided into two equal portions, where, as might be expected from its usual unsteady action, this close average of estimation was altogether wanting. In other instances, however, where, notwithstanding that every care is exercised to ensure very complete combustion, and subsequent good condensation, that the loss should occur in a *ratio so constant* is a fact not a little puzzling.

The numbers we obtain from the comparative experiments detailed above, made with the "single" jet and the "Desulphurizer," exhibit an average of increase in the quantity of sulphur arrested and determined by the addition of the latter of 32·7 per cent., being respectively, in the first set of analyses, 37·1 per cent., and in the second 28·3 per cent. It is worthy, perhaps, of remark that the gas of the second series of experiments contained the most sulphur, although the loss shown is less in degree. I have demonstrated at page 617, vol. xiv., that we incur an average of 34 *per cent. of loss* in all estimations of sulphur in coal gas, when we do not substitute the "single" jet for the "Leslie" burner in burning it as usually practised under the trumpet-tube open to the atmosphere. Doubtless a good and reliable approximative result may then be deduced, to show the advantage to be derived from replacing the usual arrangement adopted in the use of the "Leslie" burner by the "single" jet and "Desulphurizer," if we take the sum total of the following numbers:—

Superiority of "single" jet over "Leslie" burner,	
demonstrated by experiments to be .....	34 per cent.
Superiority of "desulphurizing" vessel over the	
"single" jet, also demonstrated by experiments	
to be .....	32 per cent.

These considerations prove that .... 66 per cent.  
of the sulphurous ingredients of coal gas escape estimation in analyses when the "Leslie" jet is employed as commonly applied, instead of the improved arrangement to which I have drawn attention. Let us see how this calculation is borne out, by the two experiments given in the following table.

Table showing the Results obtained by burning two equal measured bulks of Coal Gas; one of these being burned with the "Leslie" Burner, the other with the "Single" Jet, with the addition of the "Desulphurizing" Vessel.

Experiments.	Quantity of Gas analyzed.	Description of Burner.	Weight of Sulphate of Barium obtained.	Amount of Sulphur calculated for 100 Cubic Feet.
A ..	2½ c. feet ....	Leslie burner ....	1.50 .....	8.24 gr.
B ..	2½ c. feet ..	{ Single jet and Desulphurizer }	2.61 .....	14.32 gr.

Then the difference between 8.24 and 14.32 is 6.08. Further, 8.24 : 6.08 :: 100 : 73.78 per cent. loss incurred from the employment of the "Leslie" burner in the place of the "single" jet and the "desulphurizing" vessel.

If we now place these results in juxtaposition with the following experiments and remarks of M. Ellissen (vol. xiii., p. 713) we shall see that by the use of this "Desulphurizer" there is some reason to believe we really do succeed in burning and retaining nearly all the sulphur of the gas submitted to the action of the apparatus.

M. Ellissen says, "A last method of determining the amount of sulphur in gas—a method which is perhaps the most reasonable and most exact—consists in oxidizing entirely the sulphur by chlorine." He thus obtained, by the "Leslie" burner, 5.65 of sulphur; by the "oxidation" process, 10.13 of sulphur.

Then the difference between 5.65 and 10.13 is 4.48; and

$$5.65 : 4.48 :: 100 : 79.2$$

per cent. loss through the use of the "Leslie" burner.

Since becoming aware of the high determinations effected in the manner I have now described by my new adaptation, I have sought to compare them with analyses in which no sulphur present in the gas should escape estimation. My investigations upon this part of the subject, I can only now say, leave me in doubt as to whether this has yet been successfully accomplished by any experimenter. The account of them must be deferred for another communication.

The determinations given in the detail of the experiments of the next table possess much interest. They show the correctness of the view entertained at the outset of the employment of the "desulphurizing" vessel; that by burning the gas in a chamber of atmospheric air free from disturbance by draught or currents, a better or more perfect combustion would be ensured.

Table giving the Results obtained by burning several bulks of Coal Gas, in every instance divided exactly into two equal portions. The "Leslie" Burner, as commonly applied, was employed to burn one bulk at the rate of half a cubic foot per hour; the "Single" Jet (ordinary fishtail construction burner) was selected to burn the second bulk at such a rate that its combustion occupied as nearly as possible six hours.

Experiment.	Quantity of Gas analyzed.	Description of Burner used.	Weight of Sulphate of Barium obtained.	Whether "Desulphurizer" was used or not.	Amount of Sulphur calculated for 100 Cub. Ft.
First equally divided bulk {	C 2½ c.ft. ..	Leslie ..	1.31 ..	No ..	7.17 gr.
	D 2½ c.ft. ..	Single jet ..	2.60 ..	Yes ..	14.24 gr.
Second equally divided bulk {	E 2½ c.ft. ..	Leslie ..	1.82 ..	No ..	9.97 gr.
	F 2½ c.ft. ..	Single jet ..	2.19 ..	Yes ..	12.00 gr.
Third equally divided bulk {	G 2½ c.ft. ..	Leslie ..	1.46 ..	No ..	8.00 gr.
	H 2½ c.ft. ..	Single jet ..	2.29 ..	Yes ..	12.54 gr.

Now, the difference between the numbers 1·31 and 2·60 in the experiments C and D is 1·29. And the calculation

$$1\cdot31 : 1\cdot29 :: 100 : 98\cdot47$$

shows that in this case nearly *one-half* of the sulphur of the gas was lost from using the "Leslie" burner instead of the "single" kind *inside* the trumpet-tube, in the atmosphere controlled into steady inflow by passage through the pumice pieces of the "Desulphurizer." Similarly, for the bulk examined indicated by experiments E and F, the difference between the numbers 1·82 and 2·19 is ·37. Then

$$1\cdot82 : \cdot37 :: 100 : 20\cdot32$$

demonstrates that we have nearly  $20\frac{1}{3}$  per cent. loss by the "Leslie" burner in this case of comparative trial against the "single" jet and "Desulphurizer." Lastly, in respect of the difference between 2·29 and 1·46 of G H, we find, after making the calculation—

$$1\cdot46 : \cdot83 :: 100 : 56\cdot84$$

per cent. loss of sulphur arising from the same cause. This last experiment is a near approach to the average of the first two determinations C D and E F. For  $98\cdot47 + 20\cdot32 = 118\cdot79$ , the half of which is 59·39. But to add together the numbers obtained in the results of the experiments A B,\* C D, E F, G H, at this juncture is very satisfactory, because it furnishes a striking confirmation of the correctness of the conclusions deduced at page 698, vol. xv.—first, regarding the experimentally proved increased per centage average of sulphur shown when the action of the "single" jet is compared with the "Leslie" burner; and secondly, when the effect of burning the former is placed in juxtaposition with its action when governed by the transmission of the atmospheric air to burn it through the "desulphurizing" pumice arrangement. Therefore, if we take the average of the per cent. proportion of loss given in the first-made trial of the "single" jet inside the "Desulphurizer" and the "Leslie" burner brought into requisition in the usual manner, at page 698, and take the average of its addition to the average obtained in the results of the combustions of the three bulks comprised in the experiments C D, E F, G H, we get the total (from  $73\cdot78 + 98\cdot47 + 20\cdot32 + 56\cdot84 = 249\cdot41$ , which, divided by 4, gives the mean 62·35. A glance shows that the figures 62·35 is a most close approximation to the numbers, 66·00, derived from the other data of comparison at page 698, vol. xv., where the first superiority of the "single" jet over the "Leslie" burner is, to begin with, proved, together with the subsequent practical evidence of the enhancement of the "single" jet action by the addition of my pumice vessel.

If we now compare these results with those obtained by M. Ellissen in his important experiments, we shall see the justification for the remark I made at page 698, "that there is some reason to believe we really do succeed in burning and retaining *nearly* all the sulphur of the gas submitted to the action of the apparatus."

Let us now examine how this is supported by the experiments of M. Ellissen. At page 698, vol. xv. (quoted in approaching the con-

\* JOURNAL OF GAS LIGHTING, vol. xv., p. 698.



elusion of my last paper) it will be seen that he characterizes as perhaps the most exact, the method of oxidation by chlorine he put into operation to determine the sulphur, as a test of the method described by Dr. Letheby for that purpose. It proved, as will be seen upon reference, that 79.2 per cent. of sulphur was lost when Dr. Letheby's adaptation of vessels\* was, upon that occasion, taken to estimate the sulphur in a like volume of the same gas. When Letheby's apparatus was compared with that arranged by Evans, the mean of ten experiments gave the following numbers:— For Letheby's, 4.75; for Evans's, 8.37. This result is equivalent to a loss of 76.21 per cent. through the employment of Dr. Letheby's arrangement for sulphur determination. Now, three of the *direct* experiments of comparison out of the four made with my apparatus agree very closely with the determinations by complete oxidation and those with the employment of Evans's apparatus given by M. Ellissen in his report. The three results are placed in contrast underneath:—

Loss per cent. by Letheby's apparatus when compared with process of complete oxidation (Ellissen), mean of two experiments .....	79.29
Loss per cent. by Letheby's apparatus when compared with process devised by Evans (Ellissen), mean of ten experiments † .....	76.21
Loss per cent. by Letheby's apparatus when compared with my three experiments, A B, C D, G H, mean of six experiments .....	76.36

There is, then, it is proved, a very constant proportion of loss maintained when the burning action of the "Leslie" burner is compared with the above three distinct methods of estimation. M. Ellissen does not enter into a description of the complete method of oxidation by chlorine which he examined. I have applied this principle by causing the measured bulk of gas to slowly bubble through U-tubes containing an oxidizing agent of intense activity (hot hydrochloric acid and chlorate of potassium) in the attempts I have made to ascertain what we can effect towards total estimation of coal-gas sulphur by burning processes. I consider methods of combustion by some such plan of proceeding, if they can be held available, for many reasons are much to be preferred to, for instance, that of oxidation just alluded to. In the first place, an operator of considerable experience in chemical manipulation would be required to conduct the chlorate oxidation process, with any prospect of success (for it is a disagreeable experiment, and not wholly free from some danger), which will be evident further on, when I enter into an account of the way in which this is done; while the operations belonging to the much more simple system of burning the gas, as I have described it in these papers, I have often found is capable of performance by any person of moderate operative skill with the hands. I have had occasion to refer, as affording interesting results of a confirmatory character, to the apparatus devised by Mr. Evans for coal-gas sulphur estimation. I have been surprised to find many interested in the

\* *Chemical News*, vol. vii., p. 73.

† *JOURNAL OF GAS LIGHTING*, vol. xiii., p. 713.

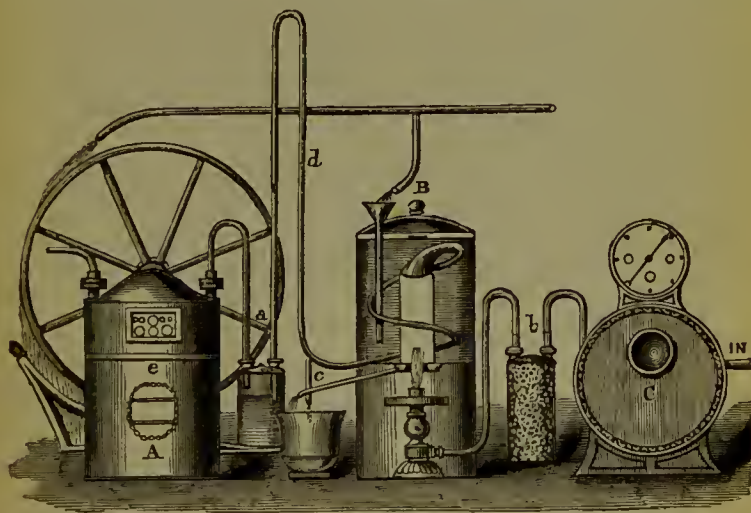
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question unacquainted with this arrangement, so I will describe it here. When I have done so, as it will be presently necessary that I should call attention to the method of determination used by M. Ellissen where the measured volume of gas is slowly transmitted over hot lime, those who have followed me through this discussion of the sulphur determination question will then have had brought under their notice every plan for accomplishing this end which has yet been suggested.

Referring to the subjoined engraving of the apparatus arranged by Mr. Evans, A is a motive-power dry meter—that is, a meter which is driven by a water-wheel or clockwork, and, by the alternate contraction and expansion of the diaphragms, it acts as a pump, drawing the air or gas in at the inlet and discharging it at the outlet. B is a condenser. In the centre of this condenser is placed a white metal chimney, similar to the 7-inch glass chimney of an ordinary Argand burner. This chimney has a funnel-shaped top, connected to a 5-8ths spiral tin pipe, arranged in coils inside



the condenser, which discharges itself at the bottom into the pipe *c*, leading through the swan neck *d* to the inlet of the meter; at the bottom of this chimney is fixed a narrow glass gallery with an outlet-pipe, for the purpose of collecting all the moisture condensed in that part of the apparatus. A Bunsen lava burner is provided with a white metal dish, through the centre of which it is made to pass, so that the point of combustion is immediately above the dish. This burner is attached to the outlet of the experimental meter C, through which, of course, the gas to be examined is made to circulate for measurement, if a graduated gasholder be not at command. Let us suppose the whole of this apparatus connected as above described, and that we wish to determine the sulphur compounds in a given quantity of gas. Then the condenser B is to be filled with cold water, which is permitted to flow through the inlet funnel-pipe to the lower part of the condenser, the water



at the surface, as it becomes successively heated, running away through the overflow-pipe. There is no necessity that this water supply for the condenser be run in at a rapid rate, for a very moderate quantity suffices to produce enough cooling effect. The small dish fastened to the burner is filled with strong ammonia; the gas ignited, and the burner then put under the condensing vessel, so that the flame may pass up the chimney. The products of the combustion are drawn through the spiral pipe, condensed there, and then flow through the small pipe at the bottom of the swan neck into a receiving vessel. The supply of ammonia to the burner is easily maintained by allowing it to drop from a small glass syphon-pipe into the dish surrounding the burner.\*

In noticing the last estimation method which has been proposed—the so-called lime mode—I should quote M. Ellissen, who, I believe, was the first chemist to apply it. “By causing,” he says, “the [measured volume of] gas to flow very slowly in a long tube filled with lime at a red heat (about 1832° Fahr.), *all the sulphur* contained in the gas combines with the caustic lime to form sulphuret of calcium and sulphate of lime. The aqueous vapour in the gas having the power of decomposing a portion of the sulphuret of calcium, it is useful [necessary] to pass the products of the decomposition into potassa, which will retain the sulphuretted hydrogen [so generated] driven off.”

There can be little doubt as to the correctness of M. Ellissen's assumption that by this process the whole of the bisulphide sulphur is capable of removal. The determinations he effected by its application, when compared with the analyses made with Dr. Letheby's arrangement, are extraordinarily, and I think, unaccountably high. To extract the results, and place them underneath, will at once show this to be the case†:—

*Comparison of the Modes of Testing the Amount of Bisulphide of Carbon in the same Coal Gas, by the “Lime Process” and the Plan given by Dr. Letheby.*

Letheby Apparatus.	Lime Process.	Loss by Letheby's Arrangement.
1 7.53 sulphur ..	9.23 sulphur ..	22.52 per cent.
2 6.88     ” ..	9.80     ” ..	42.44 per cent.
3 4.83     ” ..	43.16!   ” ..	Nearly 8-9ths.
4 3.74     ” ..	33.36!   ” ..	Nearly 8-9ths.
5 3.48     ” ..	28.00!   ” ..	Little more than 7-8ths.
6 6.31     ” ..	9.88     ” ..	56.57 per cent.

Means 5.46     ” .. 22.24     ” ..

Now, there are great and remarkable discrepancies obvious upon examining the results embodied in this table. There is no sort of correspondence between the determinations of the experiments 3, 4, and 5, and those of 1, 2, and 6. The mean of the quantities of 1, 2, and 6, is equivalent to a loss of 40.73 per cent. from the

\* See the description of this apparatus in Mr. Bannister's “Gas Manipulation,” to which I am indebted, for the most part, for this account of it.

† JOURNAL OF GAS LIGHTING, vol. xiii., p. 717.



"Leslie" burner having been used, which, from its close analogy with many results of experiments I have referred to, it is very evident is just and reasonable; but in the experiments 3, 4, and 5, where a deficiency is exhibited, in the two first instances, of nearly *eight-ninths* of the whole of the sulphur, and in the third case of above *seven-eighths*, I think we have very great reason to call in question their correctness. In fact, the application of this lime method is open to more than one serious objection besides its general tediousness. Thus, it is necessary to determine, before any result can be arrived at, the proportion of sulphur contained in the lime in its normal state, to be subsequently allowed for in the whole amount calculated from the barytic sulphate. To do this it is not quite possible to take a portion which may be considered to exactly represent an average of the whole, unless we powder the lime so finely that the transmission of the gas through the tube containing it is an impossibility. Then, again, as the whole of the lime which has been placed in the long tube to desulphurize the gas must, when the experiment is terminated, be brought into a state of solution, it is quite certain that this must entail the admixture of a large proportion of calcic salt, anything but favourable to the subsidence of the barytic sulphate from the solution when the precipitant is added. I believe, also, that it would be extremely difficult to free a small proportion of barytic sulphate deposited in a strong calcic solution of this kind, containing some thousands of grains of lime, even by copious washing with boiling water, from portions of these calcic salts, which, from what is now well known in regard to this subject, would probably, under these circumstances, adhere with great obstinacy to the precipitate. Considerations of this kind must, therefore, cause us to hesitate before receiving the determinations 3, 4, and 5, as being conclusively exact.

However, for all this, there can be no doubt that, in any case, the effect of the "Leslie" burner, when employed, as commonly done, to burn the gas, is almost always so radically bad that its use for this purpose, as I have before said, should be abandoned altogether. In one or two instances only, amongst numerous others where, with the utmost care, I have examined the consuming power of this burner, has its performance been at all satisfactory. Besides a thorough examination of the capacities in this particular of this "Leslie" burner, I have also subjected a well-made "Bunsen" burner to a similar investigation. I was surprised to find, as the experiments presently to be described will demonstrate, that its power in this respect, though much superior to the "Leslie" sort, is yet very frequently inferior to the "single" (fishtail) burner, when both were allowed to consume the gas at the rate which has been mentioned occasionally in this paper.

The following table shows the behaviour of the "Leslie" burner when placed to consume the gas *inside* the "desulphurizing" vessel. It is compared in the two experiments with the "single" jet, whose action I have said I think will be generally found to surpass all others for accomplishing the most complete combustion of coal gas, if we are to regard the conversion of its bisulphide of carbon compounds into sulphurous acid as an indication of this power.

Table showing the Results obtained by burning two equal measured volumes of Coal Gas to compare the "Leslie" Burner with the "Single" Jet, both being employed inside the "Desulphurizing" Vessel.

Experiments.	Quantity of Gas analyzed.	Description of Burner used.	Weight of Sulphate of Barium obtained.	Amount of Sulphur calculated for 100 Cub. Ft.
First { I ..	$2\frac{1}{2}$ c. ft. ..	Leslie ..	1.70 ..	9.31 gr.
bulk { K ..	$2\frac{1}{2}$ c. ft. ..	Single ..	1.61 ..	8.84 gr.
Second { L ..	$2\frac{1}{2}$ c. ft. ..	Leslie ..	2.00 ..	10.96 gr.
bulk { M ..	$2\frac{1}{2}$ c. ft. ..	Single ..	2.10 ..	11.50 gr.

In the experiments I K, the advantage is in favour of the "Leslie" burner to the extent of 5.31 per cent.; M L M, on the other hand—the "single" jet—has, as usual, the higher number, corresponding to an advantage in its favour equal to 5.00 per cent. For—

$$2 : 1 :: 100 : 5.00.$$

Although the steadier combustion is thus induced by the control exercised over its fluctuating, flickering tendency, when so enclosed in the regular ascending column of atmospheric air of the interior of the "Desulphurizer," nevertheless, even under these circumstances, as the ensuing examples demonstrate, its combustion is still liable to be very uncertain and irregular.

A bulk of gas was divided into two equal measured volumes, and both burned by the same "Leslie" burner, under precisely similar conditions, inside the "Desulphurizer."

Experiments.	Quantity of Gas analyzed.	Description of Burner used.	Weight of Sulphate of Barium obtained.	Amount of Sulphur calculated for 100 Cub. Ft.
N ..	$2\frac{1}{2}$ cubic feet ..	Leslie ..	1.50 ..	8.22 gr.
O ..	$2\frac{1}{2}$ cubic feet ..	Leslie ..	2.10 ..	11.50 gr.

Then the calculation—

$$8.22 : 3.28 :: 100 : 39.9$$

decisively proves that determinations made with the most accurate precautions with this "Leslie" burner are subject to a loss of, in round numbers, *forty per cent.*, when every condition in the experiments of comparison is observed with the most perfect regularity.

An addition to the series of experiments made with this burner is appended underneath.

Results obtained by burning two equal measured bulks of Coal Gas by the "Leslie" Burner, in the one case open to the air, and in the other inside the "Desulphurizer."

Experiments.	Quantity of Gas analyzed.	Description of Burner used.	Whether "Desulphurizer" was used or not.	Quantity of Sulphate of Barium obtained.	Amount of Sulphur calculated for 100 Cub. Ft.
P	$2\frac{1}{2}$ c. ft. ..	Leslie ..	Yes ..	2.02 ..	11.06 gr.
Q	$2\frac{1}{2}$ c. ft. ..	Same ..	No ..	1.50* ..	8.22 gr.

\* The arrangement of the apparatus in this experiment was the same as described at page 658, vol. xiv., with this exception, that the addition of the long tube *y* to the second receiver was omitted. I should mention that its employment I have found to be decidedly instrumental in increasing the unsteadiness of combustion, so difficult to obviate from irregular currents of air, especially in the "open"-air Leslie plan.

The superiority in burning power communicated to the "Leslie" burner by enclosure in the "Desulphurizer" is thus proved to be equivalent to a little under *thirty-four and a half* per cent. over the usually adopted "open"-air plan. For—

$$8.22 : 2.84 :: 100 : 34.54.$$

This ratio of superiority, it is somewhat curious to remark, coincides very closely with the relations found at page 698 to exist between the "single" jet burned under the trumpet-tube on the one hand, and inside the "Desulphurizer" on the other. The proportion in favour of the latter principle, it will be seen on reference, was then found to be 32 per cent.

The results obtained in the experiments I K, L M, P Q, if further confirmed, I consider sufficient to justify the use in analysis of the "Leslie" burner, but only in conjunction with the "Desulphurizer," to regulate its combustion. If due attention be paid to the proper adjustment of the pressure of the gas, as delivered for combustion to its orifices, I think these as high as that obtained by using the "single" jet may be possibly ensured. And, as some economy of time may be effected by the employment of the former burner, this is perhaps worthy of remark in this place.

Of the capabilities of the "Bunsen" burner for consuming the bisulphide of carbon compounds into sulphurous acid, when compared with the "single" jet and "Leslie" burner:—

I anticipated a very perfect combustion by the employment of this burner. Bearing in mind the great probability, from the experimental facts and reasoning I have adduced, that we now have at control methods by which we can estimate the whole of the sulphur of coal gas bisulphide without resorting to disagreeable and complicated methods by chlorine oxidation or lime, I have considered it well worth while to examine minutely the action of the various burners which have been hitherto employed for this purpose, before finally proceeding to describe what processes I have adopted, with the view to compare an exact system of analysis with the best results obtained by the readily performed modes of combustion, of which much description has been given in these investigations. But supposing it should eventually be found that combustion by any description of jet fails to effect the conversion of the *entire* amount of the bisulphide sulphur into sulphurous acid—for the facility with which some portions of gas obstinately resist combustion in very hot burning jets is really extraordinarily curious—are we quite justified in holding a gas company responsible for any certain proportion of this bisulphide sulphur which, under normal conditions of combustion, effectually resists burning into deleterious sulphurous compounds, from which no injurious atmospheric contamination can occur? In order to thoroughly carry out this line of reasoning, the selection of the most perfect of burners to ensure the most complete or greatest possible conversion of the bisulphide sulphur into a compound capable of entire retention, in some form of receiver which should unequivocally absorb it, has not escaped my close attention.

The following furnishes a complete inquiry into the power in this regard possessed by the Bunsen burner. As the principle upon which this Bunsen burner may not be understood



by many who may follow me in these investigations, I will describe it. The Bunsen burner in its simplest form consists of a small tube, having an orifice *a* for the emission of the required stream of gas screwed into a foot *B*; *c d* is a metal tube, fitting a long screw-thread round about *a*—as usually made, of from a quarter of an inch to half an inch internal diameter—into which the gas flows from the orifice *a*, when, by turning on the supply-tap, the gas is delivered to it through the inlet *e*. The metal tube *c d*, also round about *a*, is perforated with several holes, so that as the gas emerges from *a*, and is set on fire on the surface of the tube at *f f*, atmospheric air rushes in through these, intermingles with the gas passing out at *a*, and then causes the production of a blue smokeless flame as the gas and portions of atmospheric air burn together at the top of the tube at *f f h*. The upper part of the foot support *k g g* is generally made with a screw-thread, so deep as to permit of the tube *c d* being raised higher or lower as may be desired; at the same time allowing a more or less current of atmospheric air, by the consequent enlargement or contraction of the openings around its lower part, to flow in to suit the convenience of the operator. In this way a well-constructed burner of this kind will afford a jet of gas varying from a steady blue smokeless flame to one of blow-pipe intensity. From a mere physical examination of the kind of flame obtained by a careful adjustment of the supply of gas and the accompanying influx of air to consume it, one would be fully inclined to believe that the combustion of the gas could not be made by any contrivance of the kind more perfect. We shall see, however, as the next table proves, that this is by no means uniformly the fact, curious as such a result may appear.

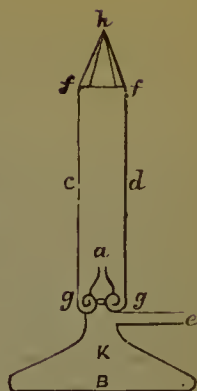


Table of Comparison of Results obtained by testing the "Bunsen" Burner with the "Leslie" Burner and the "Single" Jet.

Experiments.	Gas Quantity of analyzed.	Description of Burner used.	Whether "Desulphurizer" was used or not.	Amount of Sulphur obtained.	Quantity of Sulphur in 100 Cub. Feet.
First volume of gas, {	R $2\frac{1}{2}$ c. ft. . .	Bunsen . .	Yes . .	.437 . .	17.48 gr.
equally divided .... {	S $2\frac{1}{2}$ c. ft. . .	Leslie . .	No . .	.265 . .	10.60 gr.
Second volume of gas, {	T $2\frac{1}{2}$ c. ft. . .	Bunsen . .	Yes . .	.301 . .	12.05 gr.
equally divided .... {	V $2\frac{1}{2}$ c. ft. . .	Fishtail . .	Yes . .	.287 . .	11.48 gr.
Third volume of gas, {	U $2\frac{1}{2}$ c. ft. . .	Fishtail . .	Yes . .	.309 . .	12.36 gr.
equally divided .... {	W $2\frac{1}{2}$ c. ft. . .	Bunsen . .	Yes . .	.364 . .	14.56 gr.
Fourth volume of gas, {	X $2\frac{1}{2}$ c. ft. . .	Leslie . .	Yes . .	.431 . .	17.24 gr.
equally divided .... {	Y $2\frac{1}{2}$ c. ft. . .	Bunsen . .	Yes . .	.472 . .	18.18 gr.
Fifth volume of gas, {	Z $2\frac{1}{2}$ c. ft. . .	Bunsen . .	Yes . .	.369 . .	14.76 gr.
equally divided .... {	1 $2\frac{1}{2}$ c. ft. . .	Single . .	Yes . .	.280 . .	11.20 gr.

Experiments.	Quantity of Gas analyzed.	Description of Burner used.	Whether "Desulphurizer" was used or not.	Amount of Sulphur obtained.	Quantity of Sulphur in 100 Cub. Feet.
Sixth volume of gas, equally divided . . . .	2 2½ c. ft. . .	Single	.. Yes	.. .328	.. 13.12 gr.
	3 2½ c. ft. . .	Bunsen	.. Yes	.. .287	.. 11.48 gr.
Seventh volume of gas, containing a little bisulphide of carbon diffused in it, similarly divided . . . . .	4 2½ c. ft. . .	Single	.. Yes	.. .712	.. 28.48 gr.
	5 2½ c. ft. . .	Bunsen	.. Yes	.. .561	.. 22.44 gr.
Eighth volume of gas, with some bisulphide, also diffused, divided as last . . . . .	6 2½ c. ft. . .	Single	.. Yes	.. .671	.. 26.84 gr.
	7 2½ c. ft. . .	Same	.. Yes	.. .774	.. 30.96 gr.

With the exception of the experiments with the eighth volume, the combustion of each occupied about six hours.

Note.—In experiment 6, a very steady current of oxygen gas, transferred from a gasholder, the flow of which was exactly adjusted by being made to bubble through a U-tube of water, was conveyed into the interior of the "Desulphurizer." It was supplied during the whole continuance of the combustion.

Without now entering into a minute account of this application of oxygen gas to burn the gas (it possesses many features of great interest), or proceeding to draw any inference from the last given tabulated results,\* I will merely observe here that, seeing from the experiments upon the eighth volume of gas it appears that, no matter whether the gas was wholly consumed or not, the condensing arrangement—notwithstanding that two receivers were used—was clearly inefficient in retaining the products of the combustion, I now proceed to describe the apparatus I have devised, by which no sulphurous products can escape retention in the condensing-receivers as fast as they are generated by the burning jet.

We shall see that we shall then have the solution of the following interesting questions, which have never yet been given:—

1. What limit (if there be any) is there to the conversion of the bisulphide of carbon compounds in coal gas into sulphurous acid, when consumed by various burners in atmospheric air?

2. How can the products of such combustions be completely condensed?

I now proceed to deal with the question of the method which must be adopted to effectually arrest all the sulphurous products from the combustion of the gas burned inside the trumpet-tube. I have, for this purpose, arranged an addition to the usually employed apparatus, which we shall see completely effects this end.

It is first necessary to refer to the annexed drawing:—

\* In every determination of sulphur comprised in this table I employed a system of volumetric analysis, with a standard solution of chloride of barium—capable of being practised by a mere tyro in manipulative arts—with a great saving of time and trouble. The amount of sulphur in a solution, by this means, I can ascertain in twenty minutes; while otherwise, by the weighing system, at least thirty hours is required for the greatest accuracy. This mode I hope to give in my next communication.





M shows the large glass receiver, into which the trumpet-tube is fitted at W s, supported at a suitable height by the holder *m m m*; *z* is the pipe connected with the measured bulk of gas to be burned by the jet *p*, under the trumpet-tube *o*; *t* is the glass gutter of the "desulphurizing" vessel, which, with its pumice fragments, is further shown at *n*. The outlet *u* of the receiver condenser, instead of being associated with a second one, as drawn at page 658, vol. xiv. (L), is, by the tube bent at right angles, *x*, joined to the series of six U-tubes, *d' d d d d d*. These are likewise connected, at *e e e e e*, by glass tubes bent at right angles, passed through bored, sound, well-fitting corks. The employment of junction-tubes of vulcanized india-rubber is, of course, in this case quite inadmissible. The bottle *g* is of the wide-mouth form, attached to the last U-tube, *d'*, by the right-angled tube *e'*, inserted through the double perforated cork *h*, passing nearly to the bottom of it. In the second perforation of this cork, a short piece of glass tube, *q*, it will be observed, is fitted, the lower extremity of which only just emerges through the under side of the cork *h*. Its function is to make a junction from the tube *q* to the orifice *c* of the apparatus A B C, an elegant form of "aspirator," the office of which must now be described.

It consists of a small tube, generally made of brass or copper, open at both ends, and provided with a somewhat narrower tube let into its side, as represented in the drawing at C. The upper opening, A, is connected, by the vulcanized india-rubber tube, D, with the water-supply tap, E, over the sink, F; B, the lower outlet, is joined in a like manner to a glass tube, H, hanging perpendicularly from it over this sink. When the water-tap, E, is partially turned on (not fully, but with much care, or the water, rushing with full force into the tube, would escape both from the lower opening B and the lateral opening C), a very uniform and abundant easily controlled current of air is continuously drawn in at C, and divided into single bubbles, which are carried down with the water through the opening B, and finally pass away with it to the sink by the glass tube H. The air is thus drawn in by the suspended suction-column in this tube, and its volume to a certain extent proportional to its length.\*

The size of the "aspirator" I have found convenient for use is as follows:—The tube A B is about 1 foot in length and  $\frac{3}{8}$  of an inch internal diameter. The tube fastened into the side has an internal diameter of  $\frac{1}{4}$  of an inch. The six U-tubes are 18 inches long, with an internal diameter of  $\frac{1}{2}$  an inch, and they are each provided with a bulb blown in one limb 3 inches below the top. It is important that this enlargement into bulbs at the part mentioned be observed, as will presently be evident. The object to be attained is, that all sulphurous fumes which have not been retained by the ammonia in the large receiver M, shall be drawn for complete absorption through a solution of potassa or ammonia, placed in each of these tubes, by the action of the "aspirator." I have said that, when a carefully adjusted supply of water is conveyed from the

\* Mr. M. W. Johnson, who, 16 years ago, devised this ingenious "aspirator," states that, when this suction-column of water is allowed to run down it into a 25 feet length of tubing, air may be forced or drawn through a column of water 6 feet in height.

water-supply tap, E, to the upper opening, A, of the "aspirator," that air is continually sucked in through the orifice C: a fluid ounce of solution of potassa, or solution of strong ammonia, is a suitable quantity with which to charge each tube. When the apparatus is put together in the way shown in the drawing—the stand, K K, with its arms, being very useful for supporting the U-tubes in a steady position—in order to draw air steadily and continuously through the pumice of the "desulphurizing" vessel, up the trumpet-tube, through the condenser M, and then through the fluid of the six tubes and bottle *g*—yet with a velocity sufficient to supply atmospheric air in quantity adequate to maintain the perfect combustion of the gas by the jet *p*—it is simply necessary to join the tube *g* with C by the flexible pipe *r*, and adjust the supply of water to the "aspirator" by the tap E, until the desired rate of transmission of air be arrived at.

With the U-tubes containing the proportion of liquid named, it is necessary that the tube to contain the falling suction-column of water, H, be provided with a total length of at least 8 feet, otherwise the required rate of inflow of air cannot be properly controlled, or rendered at all times sufficiently rapid. I find it convenient, in order to possess the sink at command for getting rid of the water running away from the "aspirator," to raise this "aspirator" near the ceiling of the room. The drawing explains that this may readily be accomplished by leading a flexible tube from the water-tap, E, to the upper inlet of the "aspirator," when secured in its elevated position by fastening it to one or two nails driven into the wall of the room.

It is essential, if we would thoroughly wash the mixture of atmospheric air and unremoved sulphurous fumes by the potassa or ammonia in the U-tubes, that the "aspirator" be worked so as to maintain the alkaline solution of these in a condition of brisk agitation. Now the potassa or ammonia, from absorption of carbonic acid produced by the combustion of the gas, after a while becomes more or less changed into carbonates. The result of this is, that the solutions in the U-tubes, having worked very satisfactorily for some time after the commencement of the experiment, subsequently froth and foam in a very unpleasant manner. Rings of liquid rush up the limbs of the tubes nearest the "aspirator"—those limbs, in which by the force of the current of air the level of the fluid is raised and supported in constant agitation. If we have not adopted the precaution of setting bulbs blown in the upper part of the limbs of the U-tube in which this action continues, the rings of fluid break constantly against the cork junctions, saturating them in an embarrassing manner with portions of the solution, which it is so essential for the success of the analysis should at the termination of this operation be capable of entire removal without loss from the U-tubes. Then, again, as these rings break when they reach the cork, some of the liquid is inevitably drawn into the small tubes of connexion, *etc.* This is a serious occurrence, for in no great lapse of time the liquid in the tube next but one to the large receiver M becomes greatly augmented in bulk by the small portions of fluid of the tube placed between it and this receiver, which have been in this manner carried over from it into tube No. 2.



The force of the current of air soon carries the excess of fluid which has thus accumulated in No. 2 U-tube into No. 3; then into No. 4, and so on, until, by traversing successively from one tube to another, it finally collects in the bottle *g*. If this last vessel be provided of considerable capacity (and this should on no account be overlooked), it will safely retain any of the fluid of the U-tubes which from any cause may be carried over in this way. It is always advisable to place a bottle, fitted up as described, to intervene between the U-tubes and the "aspirator," as sketched in the drawing, notwithstanding that we have not omitted to observe that every one of the U-tubes has been furnished with the bulbs blown in them, which, in my experience, I always find effectual in preventing the contact of the enclosed solution with the corks, even when very brisk agitation of it occurs. The addition of this bottle, *g*, is a sure safeguard against the possibility of loss being incurred, if, from some want of caution in putting the "aspirator" into action, a sudden rush of air through the tubes forces portions of liquid with it into the last U-tube, *d'*, until it becomes filled, or nearly so. Then, but for the bottle *g* occupying its position in the arrangement (in which so long as the lower extremity of the short tube *g* remains out of reach of contact of the liquid carried over into it every drop of fluid collecting there will be securely held), the experiment would be completely spoiled from the loss arising by the passage of some of the alkaline solution, with its condensed sulphurous products, through the tubes *r* and *H* into the sink.

I am thus careful in describing the precautions it is absolutely necessary should be observed to avoid a useless expenditure of time and trouble by others, who, with a view to estimate sulphur in this manner, may put this system into operation, from the fact that in my first one or two attempts to apply it, I experienced a good deal of vexation through accidents arising from non-observance of the small details which I have here taken some pains to point out. If we would entirely avoid the mortification of failure, we must pay strict attention to setting up the apparatus with the safeguards I have particularized.

If potassa solution be the absorbing fluid selected for charging the tubes, in preference to ammonia—and in my earliest experiments I uniformly employed it for this purpose—as potassa which is usually kept in laboratories nearly always contain more or less sulphuric acid, one of two courses must be adopted in this case: we must either purify the solution of potassa in a way that will enable us to be completely rid of this sulphuric acid, or, what is far more convenient, prepare a few pints of the unpurified potassa solution, then take a weighed quantity of this, and pass a current of chlorine into it until no more of that gas is absorbed; boil for a few minutes, after feebly acidifying with a few drops of hydrochloric acid; filter; add sufficient solution of chloride of barium to throw down all the sulphuric acid; filter after allowing twenty-four hours for subsidence; wash dry, ignite, and weigh the precipitate thus obtained.

It is only then necessary that the potassa solution be weighed before being placed in the U-tubes. A flask is, therefore, selected which will about hold the necessary quantity of the potassa solution adequate for this purpose, and it is, with its contents, then



exactly counterpoised in the balance. When the operator has poured from it as much of the alkaline fluid as the tubes require, weights are placed upon the balance-pan until this loss of weight is made up by adding weights, which necessarily represent the true quantity of potassa solution taken to absorb the sulphurous vapours. The proportion of sulphuric acid existing in the assay of the first-weighed bulk having been ascertained, the quantity of this acid present in any second-weighed amount is, of course, then readily known by a simple rule-of-three calculation.

The experiments which follow explain how this allowance for sulphuric acid present in the potassa solution assay is easily arrived at. One-half of the bulk of gas taken to compare the results obtained by burning equal volumes of a selected sample of gas was first burned inside the "desulphurizing" vessel with the "single" jet. The arrangements of the apparatus in this case were made with every possible precaution, so as to ensure estimation of the highest amount of sulphur which could be attained by this method of proceeding; that is to say, two receivers, in which the maximum allowance of ammonia was contained, were employed for the condensation of the sulphurous products of the combustion. Six hours were occupied by this combustion, and great attention was given that the gas should burn steadily and as perfectly as could be accomplished; and in order that the gas should be consumed in an atmosphere containing much ammoniacal vapour, plenty of strong ammonia was continuously supplied to the gutter of the "desulphurizing" vessel, in place of that portion which, as it became reduced in strength by volatilization of its ammonia gas, was from time to time removed by a syphon. The weight of the sulphate of barium obtained in this instance was 1·63 grains.

The second equal half bulk of the gas was put to burn immediately the operation of consuming the first portion had terminated in connexion with the tube apparatus depicted in the drawing, Fig. 4. Into the receiver M, the same measure of ammonia as that which had been divided between the two receivers in the experiment just described, was poured before it was connected to the series of U-tubes. Then a flask, which would contain about 1500 grains of potassa solution, of specific gravity 1·27, was counterpoised upon the balance. As this flask was provided with a lipped mouth, well adapted for pouring from it without loss, no difficulty was experienced in conveying the requisite quantity of potassa solution out of it into the U-tubes. When this had been done, the flask was again placed upon the balance-pan, and the proportion added to the U-tubes ascertained by again weighing the flask. Beneath is the statement of the weight of the solution poured into the tubes:—

The flask and potassa weighed . . . . .	1436·81 grains.
After the removal of the potassa . . . . .	297·60 "

Therefore there is placed in tubes . . . . . 1139·21 grains of the potassa solution.

Then the cork connectors of the U-tubes were made good, and a junction with the receiver M fitted in the form of the tube *x*. Evidence of the perfect tightness of the corks and tubes of the

U-tubes is always afforded by the act of pressing the corks into their places. The compression of the air inside them raises and depresses the liquid contained in them to various levels. The continuance of these different levels for a short time, which always happens when good sound soft corks only are chosen, is an evident proof of the certain air-tight character of all the cork connectors of the tubes. Although no loss of sulphur could arise from an imperfect junction in this part of the apparatus, it is advisable to pay attention to securing air-tight joints, from the fact that a leakage, by allowing air to be drawn in there to supply the partial vacuum arising from the exhaustive action of the "aspirator," might seriously interfere with the required rate of inflow of atmospheric air through the pumice pieces of the "desulphurizing" vessel. When the connexion with the "aspirator" by finally joining *r* and *g* had been established, and a steady inflow of air set going by carefully putting this apparatus in action, the gas was burned off in eight hours with the same jet used with the first bulk, the gutter of the "Desulphurizer" being, as before, occasionally replenished with a little fresh strong ammonia as its contents become reduced in strength by exposure to the circulating current of atmospheric air.

When all the gas has been burned, and the products of its combustion absorbed, as it is evident they must be in one part or another of these condensing vessels, the operation of completely cleansing them of their contents remains to be performed. This, which at first glance may appear to be a troublesome undertaking, is by no means so if conducted in the following manner:—I have before described how we may proceed to effectually remove the condensed products from the receiver *M*, and the trumpet-tube *O* (see vol. xiv., page 659): also in what way the liquid of the "desulphurizing" gutter can be, without loss, added to the other portions of fluid containing the products of the analysis, so that I have now merely to deal with the removal of what is contained in the U-tubes and the bottle *g*, if, during the performance of the process, anything has been carried over from the former into the latter.

As it is scarcely possible to maintain the contents of the U-tubes throughout the process in a state of brisk agitation by the operation of the "aspirator" without causing the under side of their corks occasionally to have a small quantity of the violently shaken potassa solution thrown up or "splashed" against them, it is necessary, to begin with, that every one of these corks should be cleansed thoroughly from anything so thrown in contact by warm water. For this purpose, the right-angled tubes *e' e e e e x*, are respectively placed astride two small beakers of warm water, stood side by side, so that the two corks attached to each lower limb are placed beneath the surface of the warm fluid. After being allowed to soak for five or six minutes, they are removed, carefully shaken to free them from adherent water; the contents of the beakers added to the condensed products removed from *M* evaporating for the subsequent treatment with chlorine; fresh warm water placed in the beakers, and the cork-soaking process again repeated as before. If the right-angled tubes *e' e e e e x* then have their interiors rinsed with warm water from the washing bottle, and the cork exposure to the action of warm water once more be brought



into requisition, it is, I find, always certain that we avoid any possible loss of sulphurous condensed products through their adhering to, or passing into the interstices of, the corks. Whilst these manipulations with the corks are proceeding, the U-tubes should be placed inverted carefully over beakers to drain off their potassa solution. When the alkaline liquor has thoroughly run off, they may be easily washed quite clean by the addition, first, of a little dilute hydrochloric acid; lastly, some warm water. The warm rinsing water may be made to come in contact with every part of the inside of the tubes by closing both openings with clean sound corks, and then shaking the water inside it to and fro. The complete control over all the alkaline fluids employed to condense the sulphurous portions is completed by pouring any fluid which may have accumulated in the last vessel of the series, *g*, among the other mixed products, and, finally, after washing it clean with pure water, adding these washings likewise to the mixed products.

2.36 grains of sulphate of barium were deposited from the whole bulk of the liquids of the condenser, tubes, &c.; when thus removed they had been treated with chlorine, filtered, and mixed with chloride of barium, after careful evaporation to a moderate degree of concentration. When potassa solution is used after the transmission of the chlorine, and the subsequent application of heat, it is as well, before mixing with the barytic salt, to feebly acidify by the addition of a small quantity of hydrochloric acid.

To get at the exact quantity of sulphate of barium equivalent to the sulphur of the volume of gas examined, in this precipitate found to weigh 2.36 grains, it was only necessary to extend the examination a little further, and make the following analysis and calculation. The flask and a portion of the potassa solution taken from the stock bottle was weighed, some poured out into a beaker, and the flask again weighed. In this way it was found that 716.40 grains of potassa solution had been removed into the beaker. Although no real necessity existed for the transmission of a stream of chlorine into this weighed portion, yet it was so treated, and the rest of the details of the usual method of proceeding observed before mixing with chloride of barium to separate its sulphuric acid. Exactly .25 grain of sulphate of barium was in this way separated, after the regular allowance of time for its proper deposition from the 716.40 grains of potassa assay.

Then the calculation—

$$716.40 : .25 :: 1139.21 : .39$$

proves that the proportion of solution of potassa put into the U-tubes contained, as impurity, sulphuric acid equivalent to .39 grains of sulphate of barium.

And, further, the total quantity of sulphate of	
barium precipitated .....	equal 2.36 grains
Less .....	.39

Gives ..... 1.97 grains

as the quantity of sulphur of barium actually proportionate to the sulphur of the gas determined by the tube system.

Therefore we find the following comparative results obtained by the two methods of proceeding:—



*Table of Comparison of two Modes of Analysis followed, in which, in the first instance, the Method giving, as usually adopted (with the addition of the "Desulphurizer"), the Maximum Proportion of Sulphur was Tested against the System of Complete Condensation of Sulphurous Products of Combustion, by the addition of U-tubes of Potassa Solution.*

Experiments.	Quantity of Gas analyzed.	Description of Burner used.	Whether "Desulphurizer" was used or not.	Whether Condensing U-tubes were added or not.	Quantity of Sulphate of Barium obtained.	Amount of Sulphur in 100 Cub. Feet.
8 ..	2½ c. feet ..	Single jet ..	Yes ..	No ..	1.63 ..	8.93 gr.
9 ..	2½ c. feet ..	Same ..	Yes ..	Yes ..	1.97 ..	10.79 gr.

The three experiments, respectively numbered 10, 11, and 12, to follow immediately in detail, were made with a volume of coal gas divided into three exact equal bulks. The sulphur of the first bulk, No. 10, was estimated in every respect precisely similar to experiment No. 8 of the above table, with the two open condensers, &c. In the case of experiment No. 11, the second open condenser was removed and replaced by the six U-tubes, &c., into which 1276.70 grains of the same solution of potassa was poured to act as the perfect sulphurous absorber. In all other respects the proceedings observed in this experiment were perfectly alike to those followed in making experiment 9. Experiment No. 12 was a simple repetition in every particular (with the single exception that 915.60 grains of the potassa liquid were added to the U-tubes) of experiment No. 11. Its object was to ascertain what degree of accuracy could be attained by a comparison of results in the proportions of sulphur determined in the two estimations, for a comparative trial of this kind, it is obvious, must afford very certain evidence of the efficiency or unfitness for the purpose sought.

In experiment No. 10 the weight of sulphate of barium obtained was 1.60 grains.

In experiment No. 11 the weight of sulphate of barium obtained was 2.60 grains. The calculation—

$$716.40 : .25 :: 1276.70 : .44.$$

And 2.60 less .44 becomes 2.16—the true quantity of sulphate of barium equivalent to the sulphur of the measured volume of gas so estimated.

In experiment No. 12 the gross weight of sulphate of barium from the mixed solutions of potassa and ammonia was 2.50 grains. Following the same system of calculation—

$$716.40 : .25 :: 915.60 : .31.$$

And 2.50 less .31 becomes 2.19—the exact amount of sulphur in the gas corresponding to what had been capable of determination by the method.

Now, the difference between the quantities 2.16 of experiment No. 11 and 2.19 of experiment No. 12 (.03) is so insignificant that the issue certainly points to complete accuracy of determination,

provided perfect combustion of all its sulphur be ensured, being possible by this means. The combustion of the gas occupied in No. 11 exactly eight hours; in No. 12, nearly eleven hours. In the first the gas was not only burned with greater rapidity, but a more rapid rate of transmission of air through the apparatus was maintained from the beginning to the termination of the analysis; yet it appears that complete retention of the sulphurous fumes by the alkaline solutions was in no degree impaired by this accelerated effect.

Before placing experiments 10, 11, and 12 side by side for contrast, two more trials of the capabilities of the apparatus were made with another volume of gas as usual, equally divided under circumstances slightly modified. Instead of placing a weighed portion of potassa solution in the U-tubes, strong ammonia was added to these vessels. If strong ammonia, it was considered, would operate as a condensing material when agitated in the U-tubes as efficiently as solution of potassa, its substitution in this way must offer many advantages over the employment of the latter for the purpose. Thus the weighing of the potassa would be unnecessary, which, though a manipulation, when performed as I have described, simple and easy, nevertheless occupies some little time. Then ammonia is without difficulty obtained from manufacturing chemists perfectly free from any trace of sulphuric acid, so that the analysis of either the solution of potassa or solution of ammonia in the first instance, for after-deduction from the total determination of barytic sulphate, would again be entirely dispensed with. And, further, the addition of as much ammonia to the first condenser and the U-tube series as would suffice to well occupy the interior of those vessels, while the combustion continued, with liquid and gaseous ammonia, it was believed would thoroughly contribute to fix every particle of sulphurous acid as its vapour was successively drawn through them. To test this point, 4 fluid ounces of ammonia were added to the first large receiver M, and one fluid ounce to each of the U-tubes, in the experiment made to compare this use of ammonia, which immediately follows. At the termination of the operation the liquid in the last three U-tubes possessed a strong odour of free ammonia, and the considerable degree of cold produced in them by evaporation of some of the ammonia as the air rushes through on its road to the aspirator promotes the absorption of ammoniacal fumes brought in from the anterior vessels.

These two experiments shall be called Nos. 13 and 14.

No. 13, the first half bulk, burned in association with "Desulphurizer" and the two condensers, made to correspond precisely with No. 8, gave of sulphate of barium exactly 1.60 grains.

No. 14, the second half, burned with the addition of the U-tubes, &c., gave of the same precipitate 2.11 grains.

The coincidence of this analysis with Nos. 10, 11, and 12, is, for the success of the apparatus, as satisfactory as it is even remarkable. The gas submitted to examination in each of the eight experiments was unquestionably identical; the first bulk had been removed early in the morning from the company's main, and replaced late at night by the last bulk from the same source.

It will now be convenient to place the results of these five

experiments—10, 11, 12, 13 and 14—in the form of a table, as upon former occasions. We shall then see what degree of uniformity exists in regard to the one point having great interest to all who devote attention to this subject—that is, the proportion of sulphurous fumes which, as these experiments it will now be seen at a mere glance conclusively establish, always escape fixation in the receivers when, as they have been hitherto adapted in the process, they are always more or less open to the external atmosphere.

*Table showing Results obtained by comparing the maximum determination quantity of Coal Gas Sulphur possible, having two large Receivers containing strong Ammonia connected to the best form of Jet caused to burn the Gas inside the atmospheric "Desulphurizing" Vessel, with the Estimation effected when, instead of the second Receiver, the U-tubes, "Aspirator," &c., were employed, as has been described.*

Expt.	Quantity of Gas analyzed.	Description of Burner used.	Whether "Desulphurizer" was used.	Whether U-tubes were used.	Quantity of Sulphate of Barium obtained.	Amount of Sulphur in 100 Cubic Feet.
10	2½ c. ft.	Single Fishtail	Yes	No	1.60	8.76 gr.
11	2½ c. ft.	Same Jet	Yes	Yes, with Potassa	2.16	11.83 gr.
12	2½ c. ft.	Same Jet	Yes	Yes, with Potassa	2.19	12.00 gr.
13	2½ c. ft.	Same Jet	Yes	No	1.60	8.76 gr.
14	2½ c. ft.	Same Jet	Yes	Yes, with Ammonia	2.11	11.56 gr.

A consideration of these tabulated quantities and the calculations underneath demonstrate that the proportion of sulphurous products which are not absorbed by the ammonia of the open receivers, or become in any manner condensed there, is a very constant quantity. This is proved by taking the mean of Nos. 11 and 12, that is, 11.91 grains,

then 8.76

and  $8.76 : 3.15 :: 100 : 35.95$  per cent.

Further, the difference between 8.76 and 11.56 of experiments 13 and 14 is 2.8:—

Then  $8.76 : 2.8 :: 100 : 31.96$  per cent.

The mean of the two per centages of loss 35.95 and 31.96 is 33.95 per cent. In round numbers, consequently, the loss of sulphur through non-condensation amounts, it is decidedly proved, to *thirty-four per cent* when every precaution is taken to burn the gas slowly and steadily; to control the current of atmospheric air, on passing through the condensers, by the addition of the "desulphurizing" vessel, and thus ensure that every possible amount of condensable products be retained in those vessels. That the slight difference between the quantities 35.95 and 31.96 (3.99 per cent.) arises from any other source than a little irregularity in the burning of the gas, and not from absorption defects incidental to the action of the U-tubes, is rendered very certain by the very close coincidence in the quantities brought out in the experiments 11 and 12. No conclusion can be more safely assumed than that not a particle of the sulphurous products of the combustion of the gas



can escape being absorbed by the liquid of the U-tubes, especially if solution of potassa be placed in them for the purpose. It is easy to see, by watching the operation, that the gaseous mixture of the atmosphere and burnt products of the gas, from forming an emulsion with the fluid of every tube as they are drawn through by the action of the "aspirator," are brought into most intimate admixture with the alkaline fluid so keenly active in retaining in combination whatever acidulous vapours may come in contact with it.

Having, then, solved the problem of perfect condensation of sulphurous products generated during the burning of coal gas,\* which it will be seen, by a reference to page 819 of vol. xv., that I had proposed to undertake in the second place, I now proceed to deal with the final proposal—What limit (if there be any) is there to the complete conversion of the bisulphide of carbon compounds of coal gas into sulphurous acid, when consumed by various burners in common air? It is obvious that the solution of the first point—how the burnt products can be recovered and analyzed—can have little practical utility, unless the proportion which having escaped the burning action of the flame, because unconverted into sulphurous acid, also, as a necessary consequence, evades absorption in the condensers, can be accurately ascertained.

It appears positive, however, that uncertain and irregular burning of the gas by the use of some jets may be entirely prevented by the substitution of others. I have said that the "Leslie" jet, being by far the worst of all we can select for the purpose, must be discarded altogether. I take no credit for this detection of its shortcomings, because they were long since pointed out by M. Ellissen; I have simply confirmed the accuracy of his observations upon the point. Nothing but a series of comparative determinations of the sulphurous acid obtained with different burners would be of avail to enable us to decide upon that form best adapted to accomplish the maximum effect. The single fishtail jet unquestionably is able to do this with a degree of uniformity quite remarkable; so much so that the near approximation, all within the limits of error inseparable from analytical methods, shown in experiments Y and Z at page 698, vol. xv., page 658, vol. xiv., and Nos. 11 and 12 quoted above, communicate the liveliest interest to the investigation regarding the possibility of any sulphur escaping conversion into sulphurous acid whilst the gas is undergoing the process of burning by the jet.

The comparison of this fishtail jet with the "Bunsen" burner proves that the latter also commands great powers of combustion. A reference to the table at page 819, vol. xv., proves this to be the

\* Since making the above experiments it has occurred to me that a corroborative evidence as to the exactitude of this system could be elegantly applied in the following way:—First, set aside a volume of gas to suffice for three determinations. Then take two-thirds of it, and after equally dividing it, determine the sulphur in each; then counterpoise a small stoppered V-tube containing a few grains of bisulphide of carbon upon the balance; connect it with the interior of the holder containing the third volume of gas; when about 2 grains of the bisulphide has diffused into it remove, close with the stopper, and weigh again. The increase of sulphur determined in volume No. 3 over the proportions in volumes 1 and 2 should then exactly correspond to the quantity present in the weight of the bisulphide diffused.

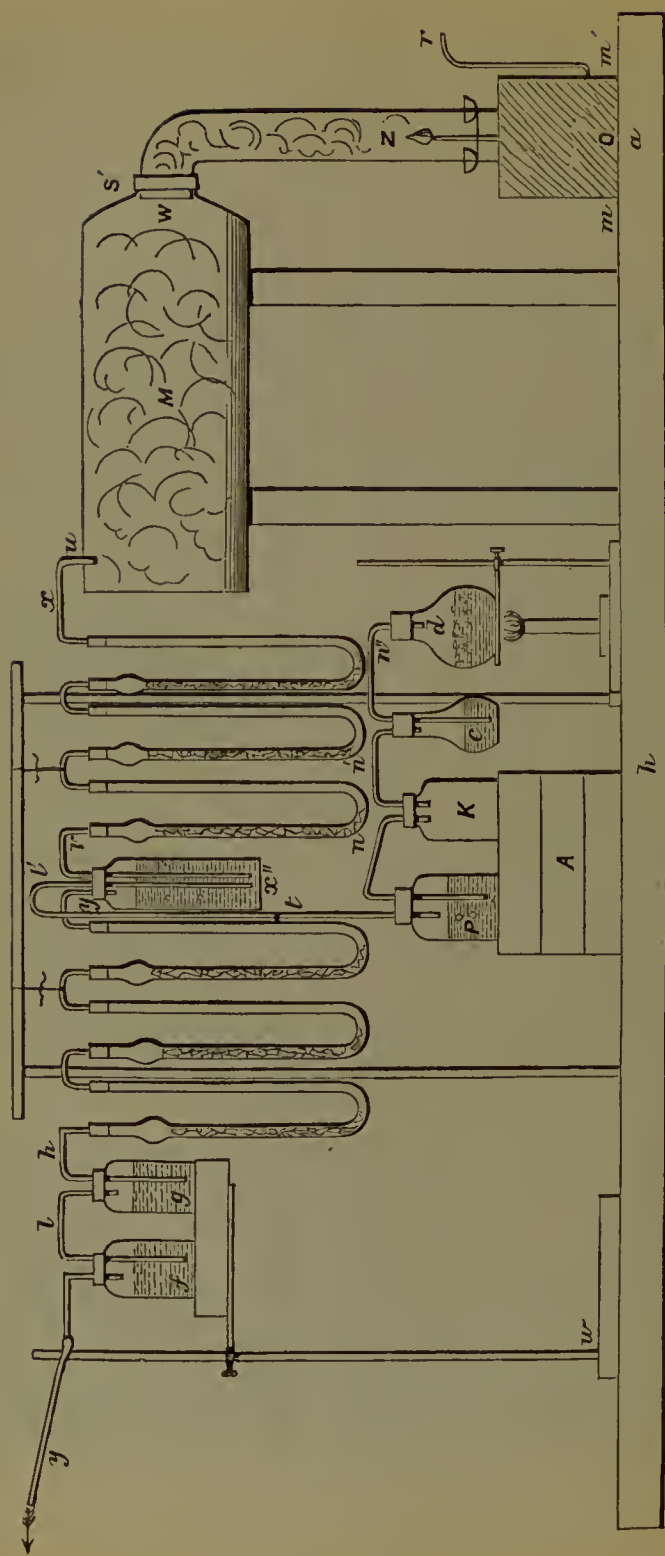
fact.\* Experiments T V demonstrate the highest estimation to be in favour of the "Bunsen" burner, for the difference between 12.05 and 11.48 is .57, and  $11.48 : .57 :: 100 : 4.96$  per cent. loss incurred by the use of the fishtail jet. Again the "Bunsen" surpasses the fishtail in U W to the amount of 17.79 per cent., for the difference between 12.36 and 14.56 is 2.20, and  $12.36 : 2.20 :: 100 : 17.79$  per cent. M Z 1 the "single fishtail" tried against the "Bunsen" lost 31.7 per cent. On the other hand, in experiments 2 and 3, the "single fishtail" has the highest estimation in its favour to the extent of 14.28 per cent. ( $11.48 : 1.64 :: 100 : 14.28$ ); and the same advantage is found on its behalf in those of 4 and 5, equal to 26.91 per cent. For  $22.44 : 6.04 :: 100 : 26.91$ . Accepting the first-quoted experiments, T V, as being in a great measure corroborative of the view that both the "Bunsen" and fishtail jets possess similar capabilities, we find this view strongly supported by comparing the mean results of the other experiments together. The mean of 17.79 and 31.70 is 24.74 per cent. superiority of the "Bunsen" over the fishtail. Conversely, the mean of 14.28 and 26.91 is 20.59 per cent. excess over the "Bunsen," obtained by the fishtail. Hence we may fairly consider the power of these two burners to closely correspond.

The apparatus represented in the figure on next page is that with which I proceeded in the next place to determine the entire quantity of bisulphide of carbon sulphur which the gas contained.

The drawing exhibits the whole arrangement, with the exception of the gasholder, in which, where a meter is not used, the required volume of gas is measured; and the "aspirator," of course, joined to the pipe *y*, is likewise omitted from it. *m a m* is the "desulphurizing" vessel, with the pipe *r* for admitting the gas to be burned by the jet *z* inside the trumpet-tube *s'*, joined to the first large receiver M. These vessels, it will be easily seen, are put together precisely as followed in the case of the first employment of the addition of the U-tubes to the receiver M, for the more perfect absorption of the burned products of the combustion of the gas. That it is, so far, quite similar and the same for a further short distance, will be evident on comparing the two engravings, until we arrive at the opening of the third U-tube, shown at *v*, and at this part of the arrangement an important variation from the first-described system is rendered necessary to effect the end desired—that of detaining every particle of the sulphur in some parts of the absorbing fluids to enable us to weigh it with precision.

Now between the third and fourth U-tubes a tall bottle is placed, as narrow in width as can be used to admit three tubes, fitted with a good close cork, through which three holes have been perforated. *x''* shows this bottle, and the U-tube, which is connected by one of its outlets to this cork, forms the junction by means of the right-angled formed tube *v*, which projects inside the bottle *x''*, nearly to the bottom of it. The smaller right-angled shaped tube *y* is fastened into the second orifice, bored through

\* The first bulk of gas in which the usual "Leslie" burner arrangement was compared with the "Bunsen" burner, combined with the "Desulphurizer," showed that 64.9 per cent. of the sulphur was lost by the use of the first-mentioned jet. This is nearly exactly in accordance with the results given at p. 698, vol. xv.





the cork of the bottle  $x''$ ; it serves to join the fourth U-tube to it, and care is taken that it does not pass more than a few lines of measurement through the under side of the cork. Then, in the usual way, this fourth U-tube is put in connexion with the fifth and sixth, while the last double-bent tube  $h$  is led nearly to the bottom of the bottle  $g$ . In the first form of adaptation this bottle only was placed in position at this part of the system; now we require a second—the bottle  $f$ —and the same shaped junction-tube  $l$  similarly connects the two together. They are held in a firm manner, standing upon the block placed upon the retort-support  $w$ , and the U-tube series are best preserved in a steady position by some such support as that figured in the sketch to which they are tied, and from which they are suspended by silk cord.

$d$  is a tolerably capacious flask, into which the ingredients employed for preparing chlorine can be promptly introduced when required. It has a good cork fitted into its neck, through which another double-bent tube,  $n''$ , passes.  $d$  is kept in a firm seat by the retort-stand ring, upon which it is supported over the Bunsen gas-burner, which must be made to gently heat the chlorine generating mixture when the evolution of that gas is needed.  $e$  is a second glass flask, furnished with a doubly-bored cork, through one opening of which the tube  $n''$  passes, as usual, nearly to the bottom, while the tube  $n'$ , in turn, fits the other hole, and continues the system of vessels by making the junction good to the glass bottle,  $K$ . But the tube  $n'$  does not pass, as with the last, nearly to the bottom of  $k$ ; on the contrary, it is merely projected a little way through the under side of the cork, fitting well into  $k$ , and  $n$  fitting another bored opening in this cork, further performs the function of uniting the interiors of  $d e k$  to the fourth flask,  $p$ , although in the case of this last the junction glass tube,  $n$ , is allowed to proceed nearly to the bottom of it. The long glass tube,  $t$ , fits the second orifice of this cork stopper of  $p$ , and at the position marked by  $t$ , a glass stopper is ground in this tube. Now, as this long tube is finally connected to the third opening, which it will be remembered was perforated through the cork stopper of the bottle  $x''$ , and passing completely through it until it nearly touches the bottom of this bottle, it will be evident that the connexion between the flasks  $d e k p$  and the bottle  $x''$  can be opened or shut off in a moment by turning on or turning off the glass tap  $t$ .  $A$  represents three blocks of wood which conveniently support the two flasks,  $p k$ .

The whole apparatus was thus joined together without vulcanized india-rubber tube being used to form any of the connexions. Then a weighed quantity of the solution of potassa was added in the usual sufficient proportion to maintain by the action of the "aspirator" a brisk agitation of the contents of all the U-tubes to each of the six of these vessels; and a further weighed portion, about equal in bulk to the measure of ammonia, always added to the large receiver  $M$ , was in this experiment added to that condenser. For it is not advisable in this case to put ammonia liquid into this part of the apparatus, as usually done. Then the bottles  $x''$  and  $g$  were filled with the same solution of potassa—likewise weighed—as high as the level of fluid indicated in them in the drawing. Care was

taken that ample air space should be afforded above the level of the potassa contained in these after it had been well saturated with chlorine gas, for an increase of volume occurs after this operation has been performed.

Before the bottles  $x''$  and  $g$  were again put in connexion with the three first U-tubes, which, as we know, had been charged with the weighed proportion of potassa, and then joined by the right-angled tube  $x$  to the condenser  $M$ , the potassa solution they contained was, in the first place, well saturated with chlorine gas. In fact, chlorine gas was transmitted through the potassa, kept quite cold by immersion in cold water, until the maximum amount of hypochlorite of potassa it was believed had been produced. The same mode of procedure was observed for the alkaline charge of the last three U-tubes, for as a duplicate set of tubes and corks for joining these together had been provided beforehand, the conversion of their alkaline liquid into hypochlorite was effected simultaneously with that of the bottles  $x''$  and  $g$ , without the necessity arising for the exposure of the cork junctions of those U-tubes to the corrosive action of chlorine gas to a greater extent than absolutely required.

When the potassa weighed solution of  $x''$ , the three last U-tubes leading from it to  $g$ , and this last bottle,  $g$ , had been thus treated with chlorine, they were placed in their proper stations, as shown in the engraving. Some water was poured into the bottle  $f$  until it occupied the level observed there. The necessary proportions of black oxide of manganese and pure hydrochloric acid, for generating chlorine gas, were, in the next place, put into the flask  $d$ . Some water, in quantity equal to the level indicated in the sketch, then was added to the flask  $e$ . The interposition of this bottle arises from the necessity that exists of, on the one hand, washing the chlorine gas as produced; and, on the other hand, preventing any injury to the course of the process from the contents of the flask  $d$ , as will sometimes happen, boiling or effervescing over into  $e$  or  $K$ . It is advisable to place the flask  $k$ , quite empty, between  $e$  and  $p$ , the last containing a very little pure water. If any very sudden absorption-effect occurs, as might happen in  $e$  or  $d$ , the experiment, after much trouble and expenditure of time, will not then be spoiled, as it otherwise would be, by the passage of fluid from  $x''$  into  $e$  or  $d$ . The bottle  $k$  (being provided of sufficient capacity) would then receive its contents, and no great inconvenience would be entailed by the operation of returning the fluid thus retained in  $k$  into  $x''$ .

From this explanation of the construction and the general character of the apparatus, I believe that most of those who have followed me through this inquiry will now perceive the efficiency of the means I have adopted to arrest all this coal gas sulphur. To do so we have simply, to begin with, to put into action the "aspirator," which, by its powerful suction effects, immediately produces a rapid flow of air through the "Desulphurizer" condenser,  $M$ , and U-tubes, into  $x''$ , where it bubbles through the hypochlorite contained there, then again through the hypochlorite of the three U-tubes and bottle  $g$ , finally emerging, after transmission through  $f$ , by the tube  $y$ , on its road to the "aspirator." When the regularity of the current is fairly established, the jet of gas  $z$  is



lighted, and replaced under the trumpet-tube. Now, all the sulphur which has become converted into sulphurous acid must be effectually absorbed and retained as sulphite of potassium, either by the potassa in the receiver M, or one or other of the three U-tubes interposed between it and the triple-tubed vessel *x''*. That being so, we have further provided, by adding to these absorbers the vessels *x'* and U-tubes, also *g*, containing the oxidizing compound hypochlorite of potassium of very great energy, for the additional oxidization of those minute portions of sulphur, which, escaping conversion into sulphurous acid from imperfect combustion of the burning jet would, but for copious contact with this active oxidizer, assuredly likewise escape, under all the conditions hitherto described, undetermined from the entire organization of condensers. To still further ensure complete oxidization, the series of flasks *d e k p* were added. Then from time to time a little chlorine could be evolved by applying heat to *d* by lighting the burner beneath it. Thus the chlorine, after being washed in *c*, passed through *k* and *p*, up the long tube *t*, and then through the hypochlorite of potassium in *x''*. Of course, all the chlorine not absorbed there must continue onward through the three succeeding U-tubes to *g*, drawn along with the current of air rushing forward to the "aspirator." I considered it advisable to possess facilities for driving a current of chlorine at command through these parts of the apparatus, as much carbonic acid is necessarily generated during the combustion of the gas, by which, if any escaped absorption in M or the first three U-tubes, but for the constant supply of chlorine, inconvenient decomposition of the hypochlorite would ensue in *x*, the second series of U-tubes, or *g*. After the disengagement of the requisite quantity of chlorine gas, the stop-cock *t* in the long ascending tube leading from *p* to *x''* was immediately shut off; otherwise, by the removal of heat from the chlorine generator *d*, or the absorption of chlorine gas in *c*, by which a partial vacuum would be produced, a rush of the liquid from *x''* would pass down the long tube *t* into *p* and *k*, and cause the embarrassment before mentioned.

The bulk of gas to be equally divided by the comparison of the two methods of analysis was, for greater accuracy, allowed to stand in the large gasholder for a week over water; then  $2\frac{1}{2}$  cubic feet were accurately measured into the gasholder, and burned off by the "single" jet with the large receiver M: six U-tubes, and bottle described above, with 2900 grains of the potassa solution placed in the apparatus (no ammonia whatever being employed) to absorb the products of combustion. The contents of the receivers were, when the experiment had concluded (it occupied seven hours), washed out with the greatest care. After treatment with chlorine, 3.33 grains of sulphate of barium were obtained.

Then the second half bulk, to distinctly decide the question of inefficient or thorough conversion of the bisulphide of carbon sulphur by the action of a gas-burning jet into sulphurous acid, was put into action. 3010 grains of potassium solution were weighed off for the absorption of the sulphur, both as caustic potassa and hypochlorite of potassium, in this second experiment. As the whole of the contents of the condenser, U-tubes, &c., would be necessarily mixed together at the conclusion of the operation, it is



obvious no necessity existed for noting the weight of that portion added to the vessel M and three U-tubes immediately adjoining, and that converted into hypochlorite for employment in "x", the three second series of U-tubes, and *g*.

When all the apparatus, corks, &c., had been washed clean, and the evaporated liquid, after the usual treatment with chlorine, had been mixed with chloride of barium, with the usual allowance of time given for subsidence of the sulphate of barium, 3.42 grains of that substance were obtained as the result.

Making the corrections rendered necessary by the sulphuric impurity of the solution of potassa employed (716.40 grains, containing, it will be seen on reference, sulphuric acid exactly equivalent to  $\frac{1}{4}$  of a grain of sulphate of barium), we get the numbers—

$$716.40 : .25 :: 2900 : 1.02$$

and  $3.33 \text{ less } 1.02 = 2.31$ —the proportion of sulphate of barium obtained by my new apparatus, without the addition of the chlorine oxidizing principle.

Further, from the second experiment, we find

$$716.40 : .25 :: 3010 : 1.05$$

and  $3.42 \text{ less } 1.05 \text{ gives } 2.37$ .

Then the calculation

$$2.31 : .06 :: 100 : 2.58$$

demonstrates that rather more than  $2\frac{1}{2}$  per cent. of the sulphurous components of coal gas of the class treated of in these papers escape during the combustion of the best form of jet available for the purpose.

Hence it would seem that no sort of combustion by a burning jet can effect the entire conversion of bisulphide of carbon into sulphurous acid. Nevertheless, the difference—2.58 per cent.—is small; almost, if not quite, I think, within the limits of error which may be fairly held as inseparable from an arrangement of apparatus certainly somewhat complicated. Further experiments (which I regret I am unable to follow out at present) will be required to firmly establish the fact as to whether the burning of the bisulphide of carbon into sulphurous acid is, under the circumstances described, as perfect as it would seem to be when subjected to the comparative test experiment in which the chlorine oxidation action is employed as a means of control. That the small amount of sulphur which, after leaving the three first U-tubes, has, by the imperfect burning of the jet, escaped conversion into sulphurous acid, should further resist complete oxidation and retention by the churning operation of the powerful oxidizing agents in the other five vessels through which it has to pass, as I have described, is not only highly improbable, but it would certainly appear utterly impossible. As the quantity of sulphur contained in a measured bulk of gas, by that mode of treatment, would thus become in great part removed from it before contact with the chlorine oxidizing fluid, by which its efficient action would be more thoroughly maintained throughout the experiment, I preferred and adopted it, as being more likely to be efficacious in determining all the sulphur than the method spoken of by M. Ellissen—that of allowing the

gas to bubble slowly through little flasks of hypochlorites of alkalies, and subsequent precipitation with the chloride of barium.

It will be instructive to compare these last two systems of analysis now with some of the others still largely used in coal gas sulphur estimations. Taking the actual increase in the amount of sulphur determined in the experiments 10 and 12, given at page 41 of this work, where the U-tube additions were made to the condenser M in place of the second open condenser, we must admit it as established that, in round numbers, 40 per cent. of sulphur may escape estimation with the best combustion of the gas ensured, *where open receivers* are employed to condense and retain the products generated.

Calculating the average of four experiments of comparison in which my addition of the "single" jet placed over the pumice "desulphurizing" vessel was tried against the arrangement recommended by Dr. Letheby, we at once obtain the following evidences of the capabilities of this latter for effecting this end. I select the four experiments, E F, G H, page 817, vol. xv., of this JOURNAL, first, because I know them to have been made with extraordinary care, and, secondly, because in ratio of loss peculiar to the apparatus they offer results the most favourable I can select for the arrangement itself.

Now the loss in E F is .....	20.32 per cent.
That of G H is .....	56.84 „

The mean of which is exactly .... 38.58 per cent.

Taking this quantity as representing the true increase of sulphur obtained by my improvements of the apparatus, we have yet to add to it that amount lost when we neglect to add the close condensing series of U-tubes. We know in that way we condense 40 per cent. more sulphur; in other words, that proportion which always gets away into the atmosphere, when open receivers are used, along with the hot column of air, even emerging from the orifice of the second receiver *u' y*, page 658, vol. xiv.

Discarding the decimals, we therefore find that the total sum of 40 and 38—that is, 78 per cent.—is the total loss when we compare the Letheby system with my just described plans of condensation and oxidization. The close coincidence with the method of "complete oxidation" obtained by M. Ellissen (see page 817, vol. xv.), in his comparative experiments made to contrast Letheby's with that principle (79 per cent.), I think I need scarcely now draw attention to; it is highly satisfactory. Then this result is not far from entire corroboration when Evans's principle was applied to test it. 76 per cent. of sulphur was the loss then shown to be caused by the Letheby plan. I, therefore, establish distinctly by these experiments the fact first announced by Ellissen, and closely confirmed by the apparatus of Evans, that by the employment of the "Leslie" jet, open receivers, &c., described in the *Chemical News* of February 14, 1863, so much used, not more than from *one-fourth* to *one-fifth* of the bisulphide sulphur of coal gas can be estimated.

I have drawn attention to a method of ascertaining the quantity



of sulphuric acid present in solution, by means of what is called the "volumetric" system of analysis—a method which may now be very largely employed with a great saving of time and labour. As from its application the necessity for collecting the precipitate upon a filter, washing the sulphate of barium—often a most tedious operation—drying, and igniting it previous to weighing, is entirely dispensed with, of course, the economy of time, and the speedy way in which we arrive at a knowledge of the proportion of sulphur in the gas—often a matter of importance in these cases—renders a description of the method of proceeding well worthy of taking a place in an essay devoted to the subject of gas-sulphur estimation.

A very small quantity of apparatus is needed for the performance of the necessary operations. A graduated glass tube is one utensil which must be provided of good and accurate construction. If it be 10 inches long and half an inch internal diameter, and graduated into 200 divisions, it is then well adapted for the purpose about to be described. A glass tube, somewhat smaller than the diameter of a quill pen, of a foot in length and drawn out at one end to a small orifice, is also indispensable. Its use is to lift fluid from the graduated tube in small quantities at a time, when that end of it with the small opening is pushed, as far as may be necessary, beneath the fluid, and the upper opening closed with the finger or thumb. If this lower orifice be sufficiently contracted, the pressure of the atmosphere will prevent a single drop from falling out of it, when, after firmly pressing the finger on the top, the tube and fluid are lifted out of the graduated vessel. Then as, of course, upon removing the finger, the liquid flows from the small lower opening, a part of it may be at any time in that way transferred in a few moments, as may be wanted, to another vessel. The liquid it contains, it is obvious, may be prevented running from it almost instantly by again restoring the finger to its former position over the upper opening of the tube, and in that way again forcing the pressure of the atmosphere to sustain it. Every drop of liquid can be made to run from its lower opening, if, after allowing it to drain out for a few seconds, the point of this lower extremity be held for another few seconds against the inside surface of the graduated tube. This is rather important, because, as it is necessary to accurately read the measure of the solution contained in it before and after the necessary quantity has been taken for the precipitating process, we must have at command a means by which we can well free the lifting tube from adhering fluid before reading off when we have completed it. It is obvious we might do so by blowing into it; but this, as moisture from the breath would certainly to some extent condense inside and dilute after a time the testing solution, is hardly advisable in application.

The standard solution of chloride of barium, I find, answers best when prepared in the following manner:—112 grains of dry chloride are dissolved in one quart of pure distilled water. Should there be the least turbidity observed after the solution is made, it must be filtered. When the solution has been thoroughly agitated it is ready for use, and may be set aside properly labelled.

To exactly value this test solution, the following preliminary



trial must be made:—The graduated tube is first filled with it; say it marks as holding 190 measures of test liquid. It is best to make a rule, in every testing experiment, of always pouring it into the tube until a certain level—say the 190 measure-mark—is reached. Then between  $1\frac{1}{2}$  to 2 grains of perfectly pure sulphate of soda (which may be readily obtained from any chemist who deals in chemical products) is taken and dissolved in about 3 fluid ounces of hot water. If a good chemical balance be used to weigh the salt, it is best to weigh it in a small cup; shake out about  $1\frac{3}{4}$  grain, then weigh again. The difference is, of course, the portion of sulphate of soda taken to assay the standard solution of chloride of barium.

I extract the following from my laboratory book regarding the fixing of the value of the last test solution I prepared according to this method. The sulphate was weighed in a small covered vessel immediately after it had been heated, so as to perfectly dry it.

It weighed first .....	147.11 grains.
After shaking out a little of the salt ....	145.52 „

Consequently ..... 1.59 grains of sulphate was taken for the test trial.

It was dissolved in a beaker containing 3 ounces of hot water, supported upon the ring of a retort-stand over a circular-shaped gas-flame of many small jets. Beaker-glasses do not crack if liquid be boiled in them when heated by a jet of this description. Then while the contents of the beaker were maintained at nearly the boiling point, some of the chloride of barium test solution, just previously poured into the graduated measure as high as the 190 measure-mark, was lifted out of it, and allowed to run into the beaker. I will now describe what always occurs. In a few seconds precipitation takes place, and after a few minutes gentle boiling, if the heat be removed, the precipitate subsides more or less completely to the bottom. However this may be, it is not very material, but the addition of a few drops of pure hydrochloric acid always expedites the settlement, and assists in the formation of a much clearer supernatant liquid than can, without its employment, be ensured. It is impossible to ascertain whether sufficient chloride of barium has been added by dropping small portions directly into the beaker-glass. But we can arrive at this important truth with facility in the following manner:—A second lifting-tube must be provided of convenient length to suit the height of the beaker-glass, nicely rounded at the top opening, to admit of ready closing by the pressure of the finger. Two small very clear glass test-tubes (3 inches long and rather less than  $\frac{1}{2}$  an inch diameter are the best size) are then by means of this lifting-tube about half filled with the liquid of the beaker lying above what sulphate of barium has precipitated, after which the lifting-tube is placed over the beaker to drain; then two or three drops of the test solution are added to one of the test-tubes, and if *increased turbidity, on comparison with the liquid of the second test-tube* be remarked, both are emptied back into the beaker, rinsed clean with hot distilled water, which is also added to the beaker, and set aside for the next repetition of this process. Then more liquid of the graduated tube is transferred to the

beaker, the gentle boiling repeated as before, subsidence for a few minutes permitted, some of the liquid once more removed by the lifting "pipette" belonging to the beaker (after once or twice rinsing it out with the supernatant liquid to ensure the removal of the remains of the fluid of the preceding test trial) into the test-tubes, and the test of increased turbidity examined, as before, by remarking accurately any distinction which, in this regard, arises upon the addition of the testing chloride. A little practice enables us to hit without difficulty the exact point when the last drop or two from the "pipette" ceases to produce the slightest precipitation; and more than this, the same amount of experience soon makes known to the operator the exact quantity necessary to complete the precipitation, formed from the judgment being guided by the degree of turbidity observed after one or two comparisons of the test-tubes.

Now the 1.59 grains of sulphate of sodium required, I found, exactly 100 measures of the barytic solution for complete precipitation.

The measure of accuracy at command, after a little practice, is demonstrated by the second experiment of control now to be detailed:—

Vessel and sulphate weighed .....	145.52 grains.
After shaking out some into cooling beaker	143.82 "
Quantity taken for the assay .....	1.70 grains.

This quantity was precipitated by the addition of 108 measures of the test solution.

Now the calculation

Equiv. of Sulphate.	Equiv. of Sulphur.	Taken Sulphur.
71	: 16	:: 1.59 : 35

to which 100 measures of the standard solution are equivalent.

Again, 71 : 16 :: 1.7 : 38 of sulphur, to which 108 measures of the test solution are proportionate.

Then the calculation that

$$.35 : 100 :: 38 : 108$$

clearly demonstrates the high degree of exactitude between the experiments and the calculated quantities of the two trials.

One more example will serve to show the application of the method to a determination of the sulphur in  $2\frac{1}{2}$  feet of coal gas. The concentrated liquors, after the chlorine treatment in substitution for the nitric acid acidulation, was filtered into the test beaker, gently boiled, and the test barytic solution as described run in by the "pipette" until it was found that 130 test measures had been added.

Then the calculation

Measures.	Sulphur.	Used.
100	: .35	:: 130 : .45 of sulphur

contained in  $2\frac{1}{2}$  cubic feet. Then simply to multiply by 40 gives the proportion of sulphur for 100 cubic feet of the gas; in this instance, therefore, amounting ( $.45 \times 40$ ) to 18 grains. The preci-

pitae formed in the beaker when ammonia has been used to absorb the products of the combustion of the gas always subsides very well, from the consequent presence of chloride of ammonium in the solution. There is no necessity whatever for any acidification with hydrochloric acid in such a case, but it must not be overlooked in preparing our solution for the assay test. If these precautions are duly attended to, the whole process may be completely performed in from twenty minutes to half an hour.

I may close this series of examinations by the following summary of my results:—

1. That in determining the sulphur of coal gas, the "single" (fish-tail) burner caused to consume the gas at the rate of  $2\frac{1}{3}$  cubic feet in five to six hours effects its combustion most completely. That even under these circumstances  $2\frac{1}{2}$  per cent. of its sulphur cannot be burned into sulphurous acid.

2. That in no case can the sulphurous products of the combustion be wholly recovered where condensing receivers open to the external atmosphere are employed. The best arrangement of apparatus set up on this principle loses 40 per cent. of sulphur. And the arrangement given by Dr. Letheby, I find, from the same cause, always entails a loss varying from *three-fourths* to *four-fifths* of the bisulphide sulphur of the gas.







